University of Southern Queensland

Faculty of Engineering and Surveying

Project Title: Dielectric, thermal and mechanical properties of sawdust reinforced epoxy composites post-cured in microwaves

A dissertation submitted by

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ABSTRACT

To process composites, post-curing with heat is often required in order to achieve the desired heat distortion temperature (HDT). Post-curing the composites with conventional oven would often take 6 or 8 hours. However, if the composites are post-cured in a microwave oven, it only requires a few minutes. Hence, the aim of the project is to investigate the suitability of microwave for material processing. Epoxies are polyethers and they are widely used to produce coatings, structural adhesives because they have excellent adhesion and low cure shrinkage. Epoxies are also good dielectric and have been used to manufacture printed circuit boards (PCB).

The main objectives of this project are:

- To produce specimens with different percentage by weight of sawdust.
- To measure the loss tangent and dielectric constant of the specimens.
- To measure the tensile strength of the specimens.
- To measure the glass transition temperature of the specimens
- To consider both of the material costs and the test results to recommend the best specimens for different industrial applications.

This report has found that the sawdust reinforced epoxy resins have higher Young's modulus, higher dielectric constant but lower tensile strength and yield strength. The loss tangents of the sawdust reinforced epoxy resins were slightly higher than the loss tangents of pour epoxy resins. Hence, the sawdust reinforced epoxy resin is slightly more efficient to be post-cured in microwave than pure epoxy resin. This report has also found that oven cured epoxy resins have the lowest dielectric constant, the lowest loss tangent, and the highest glass transition temperature. Hence, oven cured epoxy resin is the best candidate for making PCBs.

Acknowledgements

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I also would like to thank Mr Mohan Trada for assisting me with the microwave post curing. His safety advice and expert instructions were invaluable in helping me to successfully compete this project.

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Nomenclature

Glossary of Terms

TM - Transverse magnetic wave

TE - Transverse electric wave

TEM - Transverse electromagnetic wave

HDT - Heat distortion temperature

DMA - Dynamic mechanical analysis

PCBs - Printed circuit boards

1. INTRODUCTION

1.1 Introduction

Composites have the great flexibility of being able to be modified with their material properties such as electrical, mechanical and chemical. Hence, they are being increasingly manufactured to suit different applications in different engineering industries. To process composites, post-curing with heat is often required in order to achieve the desired heat distortion temperature (HDT). Post-curing the composites with conventional oven would often take up to 6 or 8 hours. However, if the composites are post-cured in a microwave oven, it only requires a few minutes. This significant reduction in production time means increased productivity and therefore more profit for the manufacturers. However, there is a major problem in using microwave to heat up the composites. That is the microwave oven cannot heat up composites evenly by itself. Uneven heating produced inside the material would cause cracks or distortion to emerge in the object which would greatly compromise the mechanical strength of the material. This project will demonstrate the advantages and capability of the microwave oven in material processing by producing the well post-cured composites.

1.2 Project Objectives

The aim of this project is to produce a range of epoxy resins with different percentages by weight of sawdust as filler. The result of this project is to demonstrate the effect on dielectric, thermal and mechanical properties of the final product by adding different percentage of sawdust and the cost reduction by using cheaper filler such as sawdust. The objectives of the project are:

1. To research information about tensile properties of sawdust filled epoxy resin and how they are affected by microwaves. This will help to develop a comprehensive understanding of the physical principle behind microwave heating, and the understanding of the bonding mechanisms of composite materials.
2. To produce specimens with different percentage by weight of sawdust.

3. To post-cure the specimens with microwaves.
4. To conduct a test to measure the loss tangent of the specimens. This test would help to determine the suitability of the composites to be post-cured in microwave oven.
5. To conduct a Dynamic Mechanical Analysis (DMA) test to measure the glass transition temperature.
6. To implement tensile test on the specimens by following the instructions of AS1145.4 (2001).
7. To analyse the test results.
8. To perform a cost analysis, that will provide evidence to prove sawdust filled composites are more cost effective in comparison to the pure epoxy resin products.
9. To consider both of the material costs and the test results to recommend the best specimens for different industrial applications.
10. To suggest possible solution to minimise defects.

1.3 Environmental implications

To protect the environment, the disposal method at the end of the life cycle of the products should be seriously considered. It is better to develop some methods that can decompose the epoxy composites other than a simple landfill.

1.4 Safety

Similar to other electrical equipments, there is a potential hazard for electrical shock due to both the insulation failure of the components inside the equipment, and the earthing connection failure.

Many people misperceived microwave like other ionizing radiation sources such as x-ray can cause genetic mutation. Meredith (1998, p. 22) clarified microwave radiation is

not an ionizing radiation. It has the effect on human bodies no more than producing heat.

According to Australia Standards AS60335.2.25 (2002, p26), the microwave leakage from the oven should be less than 50 W/m^2 at the distance of 50 mm from the external surface of the oven.

Epoxy resin and hardeners have moderate chemical hazards to human body. Vapors of epoxy can cause allergic reaction to human lungs. People who breathe in these vapors in long term can develop occupational asthma (California Department of Public Health 1989).

Skin contacts with epoxy resin and hardener can cause skin reaction such as redness, swelling and itching on the area of contact, these symptoms could continues for a few days then disappear. The most dangerous situation is the resin or the hardener contacted with eyes, as these chemicals can cause severe damage to the eyes.

To avoid the potential chemical hazards, people should ensure a good ventilation condition for the working environment. Gloves, goggles and masks should be worn during the experiment. According to the manufacturer's (ATL composites Pty Ltd) manual, in the occurrence of eye contact, the eyes should be immediately flushed with running water for a minimum of 15 minutes and then seek medical advice. Also if people accidently swallow resins, please don't try to induce vomiting and immediately seek medical assistance. If people swallow hardeners, also do not induce vomiting, and drink plenty if milk or water and seek professional medical advice. Furthermore, if skin contact happened, the contaminated clothing should be removed and the affected area of the skin should be washed with ATL's 845 hand cleaner and running water.

In addition to the project specified personal protection equipment, fully enclosed footwear, and long sleeve shirt should also be worn to provide better protection to against the unpredicted accidents. As a good practice for conducting experiments, the

bench should be always kept clear and clean. Do not bring food and drinks into the laboratory.

1.5 The Resource analysis

In this experiment, the requirement is to mix the resin with three different sizes of sawdust. The required sizes for sawdust are 300 μm, 425 μm and 1180 μm respectively. For each mixture of different size sawdust, three different specimens with different percentage by weight of sawdust which are 5%, 10% and 15% are required to be produced. Therefore there are total nine specimens need to be produced to complete this project. Each sample required 420 g R246TX epoxy resin and 105 g H126 hardener, multiply these numbers by nine, the total epoxy required for the experiment is 3780 g and total hardener is 945 g. The experiment also required 217.21 g of 300 μm, 425 μm and 1180 μm sawdust for each. The sawdust has been acquired from Toowoomba Timber Mart which is a local timber mill, free of cost. In order to separate sawdust into required sizes, three corresponding sifters which are provided by the Centre for Excellence in Engineered Fiber Composites (CEEFC) are used. The epoxy resins and the hardener are purchased from ALT Composites Pty Ltd. The funding for purchasing the resins, hardeners, and microwave oven and personal safety equipments is provided by University of Southern Queensland. Three 30 cm by 20 cm alumina trays were purchased from BigW Supermarket as the casting moulds. The tensile test equipment and the instrument to measure the dielectric property of the materials were provided by USQ.

2. LITERATURE REVIEW

2.1 Epoxy resins

Epoxies are polyethers and they are widely used to produce coatings, structural adhesives because they have excellent adhesion and low cure shrinkage. Moreover, Epoxy resins are also excellent dielectrics and heat conductors. These properties make them an ideal material for making printed circuit boards. As the epoxy resins often have smooth finishing, they are also used in making the rotor blades of wind turbines. In addition, epoxy resins are also extensively used to make aircraft,, oil rigs, chemical storage tanks and high speed boats because of their outstanding chemical resistance.

Epoxy resins usually exist in two states. The liquid state of epoxy resins are mainly for storing purpose. When the epoxy resins mix with the hardeners, the chemical cross links would be formed between these two chemicals and the mixtures will turn into a hard solid state. This hardening process is called curing, and it is irreversible. It is quite often to find that there are some of the chemicals in the mixture remain unreacted. The extent to which the curing reaction approaches completion is called the degree of cure (Pritchard 1999, p.13). This incompletion of the chemical reaction would affect the physical properties of the composites. Hence, in order to facilitate the reaction to approach 100% completion, it often requires to further process the composite by heating it up in several steps. This process is called post-cure. Post-curing can improve the moisture resistance and physical strength of the composites, but excessive post-curing could increase the brittleness of the composites and the risk of decomposition of the composites.

2.1.1 Types of epoxy resins

There are many members in the family of epoxy resins. One of the important members is the diglycidylether of bisphenol A (DGEBA). It has been the popular choice to the industry because of its fluidity, physical strength after curing and cheap purchasing

price. On the other hand, it has relatively low value of Tg less than 120^{o}C. Furthermore, the cycloaliphatic epoxies which are also important members of the family have extraordinary arc-track resistance to against the high voltage breakdown. Hence, cycloaliphatic resins are used to cast the high voltage bushing. Figure 2.1 shows typical high-voltage bushings made from cycloaliphatic epoxy busing.

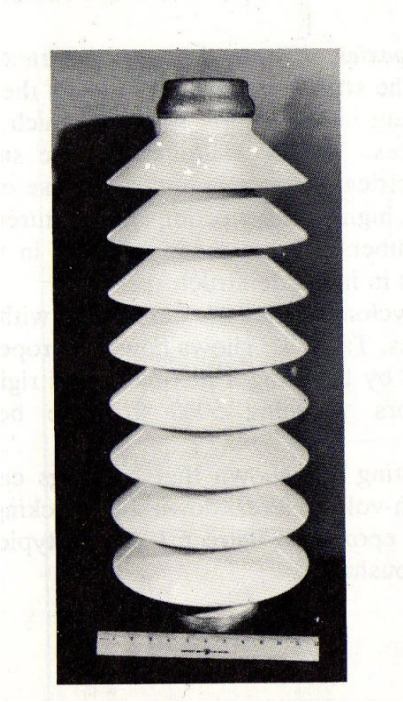


Figure2.1: High-voltage bushing cast from cycloaliphaatic epoxy (Harper 1992, p. 6.14)

There are several hardeners available in the market. One of the widely used hardener is aliphatic amines. Comparing with other hardeners, aliphatic amines require lower curing temperature. Aliphatic amines, such as diethylenetriamine (DETA), dimethylaminopropylamine and diethylaminopropyllamine, can cure epoxies at room temperature. However, these chemicals tend to absorb the moisture from the air, and hence slow down the curing process. Another disadvantage of this hardener is that there will be a large amount of heat released from the chemical reaction between the epoxy resins and the hardener. Hence, aliphatic amines are only used to cure small amounts of epoxy resins to avoid overheating the composites. Another type of hardener is aromatic amines. Aromatic amines include metaphenylenediamine (MPDA) and methylene dianiline (MDA) are solid at room temperature. The composites which are made from

epoxy resins and aromatic amines have the highest physical properties in the epoxy system. However, in recent years, aromatic amines has been found to be a carcinogen (Harper, 1992), thus the use of aromatic amines is prohibited. Moreover, some acids and anhydrides are also used as hardeners to cure epoxies. The composites made from epoxy resins and acids and anhydrides can be used for high temperature applications.

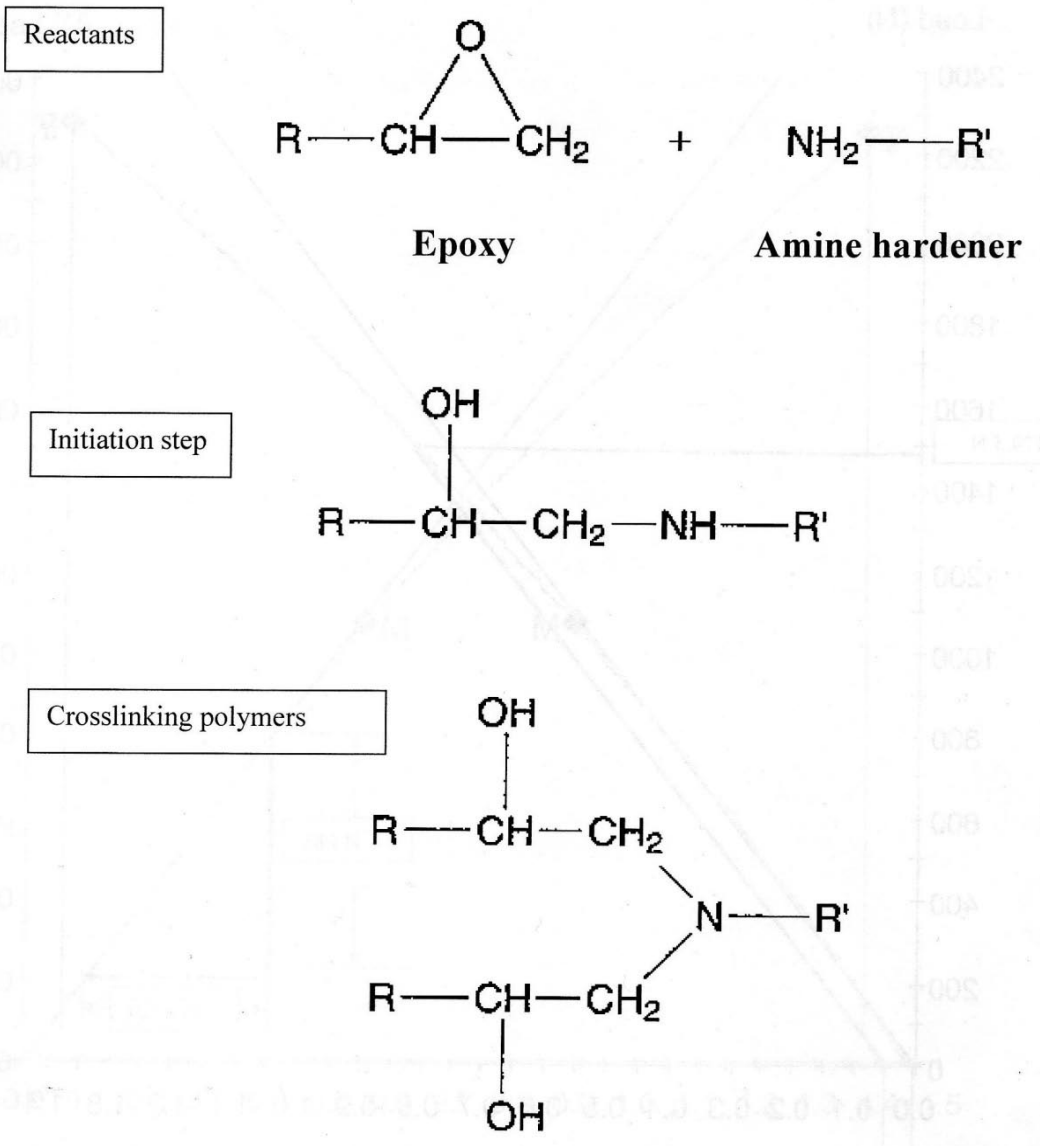


Figure2.2: Schematic of crosslinking of epoxy using amine harder (Ku 2000 et al.)

Figure 2.2 demonstrates the chemical reaction taking place between epoxy resins and hardeners. The epoxy at the top left corner of Figure 2.2 represents a molecule of the DGEBA. The amine hardener at the top left corner represents a molecule of DETA. As

shown in Figure 2.2, every molecule of amine hardener has two reactive hydrogen. Each of the hydrogen will react with one epoxide group, hence two epoxy molecules are bound together via the hardener. This process is known as cross-linking. A distinctive advantage of this type of polymerization is that the hardener becomes part of the composites, and there are no products of condensation being produced. Since the physical size change on this type of polymerization is minimal, the shrink rate is minimized. High shrinkage could lead to the distortion of the final product and hence reduce the physical strength of the final product.

2.3 Fillers

Fillers are often added into the epoxy composites to modify or improve different properties of the composites. For example, carbon fibers as fillers can increase the chemical resistance of the epoxy composites. Glass powder can increase the moisture resistant of the epoxy composites. The variety of sawdust used for this project can increase the physical strength of the epoxy composites. Metal powders, such as aluminum, silver and copper, can increase the electrical and thermal conductivity of the composites. Fillers normally do not have chemical reaction with the epoxies. To be considered as good fillers, they should stay in suspension in the mixtures and have low cost.

2.4 Glass transition temperature

Glass transition refers to a polymer undergoes a reversible transformation from a brittle and rigid glass-like state to a viscous rubber-like state. This transition usually takes place over a range of temperature (Harper,1992). The glass transition temperature T_g is the temperature in the center of the range. When the temperature is lower than the glass transition temperature, the material would become glassy. On the other hand, if the temperature is higher than the T_g, the material would tend to be rubber like. The change of the physical states will result in rapid change of the physical, mechanical, electrical and thermal properties of the materials. The content of moistures can greatly reduce the

value of Tg. Harper (1992, p.21) motioned that for each 1% moisture absorbed by the epoxy resin, the T_g will be lowered by 20^oC. On the other hand, the glass transition temperature can be increased from the cross-linking reaction, as the reaction terminates the free rotation of the end groups.

2.5 Moisture absorption of resins

Resins often tend to absorb moisture from air. As stated by Pritchard **(**1999), there are four factors can affect the moisture absorption of resins. The four factors are the polarity of the molecular structure, degree of crosslinking, degree of crystallinity and degree of curing. The degree of curing usually indicates the number of monomers (resin residuals) and hardeners. A recent study also found a rapid raise in temperature could enhance the moisture absorption of epoxy resins. Figure 2.3 shows the result of the moisture absorption study. The experiments carried out with several different resins in a wet environment. The temperature raised from 50 ℃ to 200℃, and most of the under test specimens reached the their maximum of moisture absorption at 150 ℃ , then continually fell down as the temperature increased.

The increasing moisture content results in reduction of glass transition temperature of the epoxy resins. Table 2.1 shows the relationship between the moisture content and the glass transition temperature. As shown in Table 2.1, when the moisture content increased from 0% to 1.26 % by weight, the primary glass transition temperature decreased from 222 ℃ to 198℃. One possible explanation for the moisture absorption enhancement is that the sudden change in temperature could create microcracks within the body of the samples. Once the microcracks existed, the moisture content tends to invade into these tiny spaces. In this project, the process of post-curing in microwave was originally carried out with a glass of water placed in the microwave oven to prevent the sample from overheating. However, the consequence of reconsidering the effect of moisture content on glass transition temperature discussed above, the samples were reprocessed in the microwave oven with the glass of water removed.

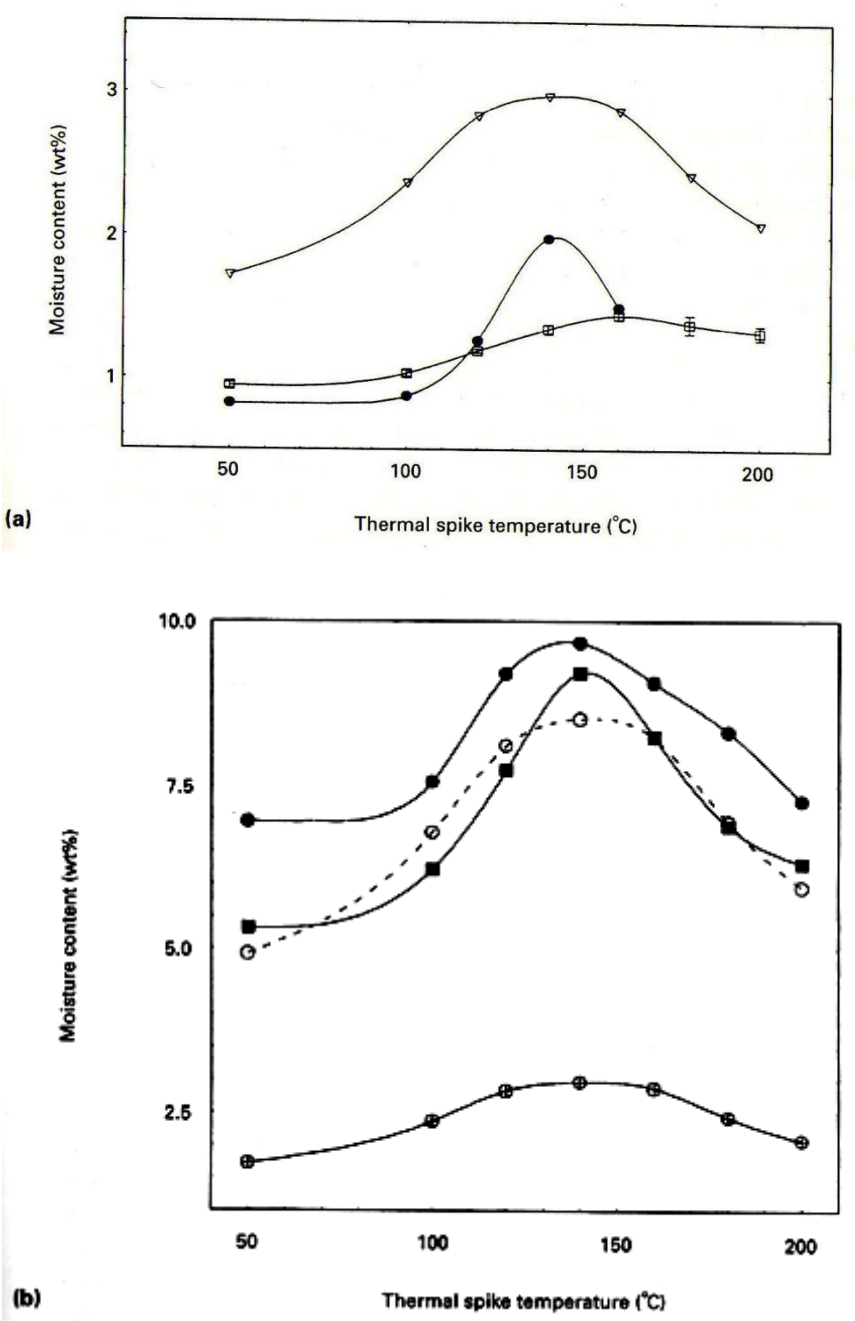


Figure 2.3 (a) Moisture enhancement effect on thermal spiking for a series of carbon fibre unidirectional laminates; ●, Narmco Rigidite 5245C; □, Fibredux 927 and ∇, Fibredux 924C. (b) Moisture enhancement on thermal spiking for a range of matrix resins; ● base epoxy resin-924E; ■, thermoplastic modified epoxy matrix resin-924T; ○, calculated matrix and composite-924C (normalised); and ⊕, 924C composite. (Pritchard 1999, p.87)

Table 2.1: Effect of thermal spiking and moisture condition on the primary (Tg1) and secondary (Tg2) tanδ peaks for advanced epoxy resin system (Pritchard 1999, p.90)

Spike temp (°C)	5245C O_{16} laminates			927 O_8 laminates			924 O_8 laminates		
	H_2O content (wt%)	T_{g1} (°C)	T_{g2} (°C)	H_2O content (wt%)	T_{g1} (°C)	T_{g2} (°C)	H_2O content (wt%)	T_{g1} (°C)	T_{g2} (°C)
dry	0	222	–	0	223	–	0	234	—
control	0.82	200	174	0.98	198	183	1.72	222	179
100	0.87	205	172	1.03	197	172	2.37	216	155
120	1.26	198	157	1.19	196	162	2.84	216	151
140	1.98	200	146	1.34	197	152	2.98	216	152
160	1.50	200	124	1.44	190	140	2.88	215	150
180				1.38	195	126	2.43	216	152
200				1.32	196	125	2.08	216	153

2.6 Yield strength

In materials science, the elastic limit is an important characteristic of material property to be measured. Before this limit, the deformation results from the applied force will disappear and the material will restore to its original dimension when the force is removed. However, once the force applied to the body of a material is beyond the elastic limit of the material, the permanent deformation so called plastic deformation will occur on the object's body, and this deformation is irreversible. Yield strength is the strength at the point which the plastic deformation just happened. Yield strength can be calculated by the equation as follow:

$$\text{Yield strength} = \frac{Yield\ load\ (F)}{Original\ cross\text{-}sectional\ area\ (A)} \quad \text{(MPa)} \qquad (2.1)$$

2.7 Tensile strength

Tensile strength is the maximum stress a material can withstand. After the applied force passes this point, the material may be broken by the force at anytime. Tensile strength can be calculated by:

$$\text{Tensile strength} = \frac{Maximum\ load\ (F)}{Original\ cross\text{-}sectional\ area\ (A)} \quad \text{(MPa)} \quad (2.2)$$

2.8 Young's modulus

Young's modulus E is defined as the ratio of stress σ to strain ε. It is the measure of the stiffness of the material within the elastic limit. The extension of the material increases linearly with the load increases before the load reaches the elastic limit. Stress σ is the average force per unit area of an area which the internal force applied to, thus Stress σ = Force F/Area A, its SI unit is Pa. strain ε is the ratio of the deformation in the direction of applied force to the original length of the material, therefore ε = deformation ΔL/original length L. By combining these equations, fields the equation to calculate Young modules:

$$E = \frac{stress}{strain} = \frac{\sigma}{\varepsilon} = \frac{\frac{F}{A}}{\frac{\Delta L}{L}} \quad \text{(MPa)} \quad (2.3)$$

2.9 Microwaves

2.9 .1 Microwave fundamentals

Microwaves have been widely used in the modern world. The most well-known applications include radars, GPS and microwave ovens. This project will focus on its application in the area of material processing. As mentioned by National Research Council (NCS) (1994, p.9),the typical microwave frequencies for materials processing are 915MHz, 2.45GHz, 5.8GHz, and 24.124GHz.

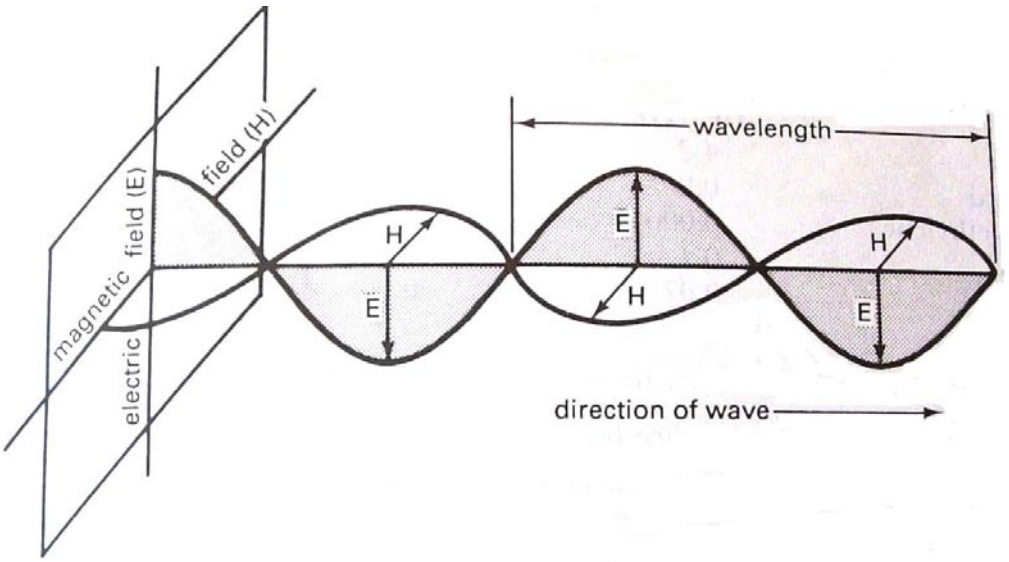


Figure 2.4: Electromagnetic wave representation (Platts 1991, p.5)

Microwaves are electromagnetic (EM) waves which have the frequency bandwidth from 300 MHz to 1000 GHz (Sadiku 2001, p.638). Figure 2.4 describes the basic representation of EM waves in free space. E is the electric field strength and H is the magnetic field strength. E and H have an inherently interactive relationship between them. This relationship can be best explained by Maxwell's equations:

$$\text{Curl } E = -j\omega\mu H \qquad (2.4)$$

$$\text{Curl } H = (\sigma + j\omega\varepsilon)E \quad (2.5)$$

Where μ is the permeability, ε is the permittivity and σ is the conductivity of the medium respectively. Maxwell's equation can be simply explained in such way that if the electric field strength E varies with time, it would generate a magnetic field and vice versa.

2.9.2 Microwave interaction with matters

The main mechanism for microwaves to interact with materials is to polarize the materials. NRC (1994, p.32) indentifies that there are three ways the electric field of the microwave can cause polarization in the materials. Firstly, the electric field can distort the electron cloud of the single atom (due to the random movement of the electrons around the nucleus), especially to the most outer electrons as they receives less attraction force from the nucleus. Secondly, some of the molecules have asymmetrical electrical charge distribution known as dipoles, although the molecules as a whole are neutral. When an electric field exists near the molecules, the electric force would drag the dipoles to align with the field. The heat generated on dielectric is due to the induced dipole moment or the bonding distortion caused by the applied electrical field. Through this process, the energy is stored in the electrical field to be converted to the kinetics energy of the molecules of the dielectric and thus increase the temperature of the dielectric.

In order to understand the effectiveness of the microwave energy to be absorbed by dielectrics, some of key material characteristics of dielectrics need to be introduced here. These characteristics are the complex relative permittivity $\varepsilon_{r,}$ the dielectric constant ε', the loss factor of the dielectric ε'' and the loss tangent tan δ. The complex relative permittivity is defined by the equation:

$$\varepsilon_r = \varepsilon' - j\,\varepsilon'' \quad (2.6)$$

The loss tangent is defined as:

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \qquad (2.7)$$

The loss factor ε'' varies not only with frequency but also with temperature, moisture content, physical state and composition.

Platts (1991, p.10) points out the reflection of microwave results at the air to material interface is proportional to the dielectric constant ε'. The larger the dielectric constant of a material is, the more reflection results when the incident wave hits the object. This effect could be explained by studying the reflection coefficient ρ. The reflection coefficient ρ is the ratio of reflected wave to the incident wave. The relationship between the reflection coefficient and dielectric constant is described by the equation (Ku et al.1998):

$$\rho \approx -\frac{(\sqrt{\varepsilon'}-1)}{(\sqrt{\varepsilon'}+1)} \qquad (2.8)$$

As $\sqrt{\varepsilon} = \frac{c}{v_p}$ where c is the speed of electromagnetic wave in a vacuum, and v_p is the speed of electromagnetic wave travelling inside the dielectric. As nothing can be faster than the speed of electromagnetic wave in a vacuum, so ε' must be a value greater or equal to 1. As shown in Eq. (2.8), if ε' equal to 1, ρ will be equal to 0, which means there is no waves to be reflected at the interface. Moreover, if ε' approaches to infinity then ρ will be equal to -1 which means all of the waves are reflected at the interface. By inspecting these two extreme conditions, it can be concluded that the reflection results at the interface will increase, as the dielectric constant ε' increases.

After the microwaves enter the dielectric materials, the skin depth D become the key parameter to measure the effectiveness of microwave to penetrate the materials. The definition for skin depth is the depth from the surface to where the electric field strength has dropped to 1/e (note: e is the natural logarithm) times of its original field strength (Ku et al.1998). The skin depth can express as:

$$D \approx \frac{2}{\omega\sqrt{\mu_o \varepsilon_o \varepsilon'}\tan\delta} \text{ (m)} \qquad (2.9)$$

Where ω is the radian frequency of the microwave;

μ_o is the permeability of free space;

ε_o is the permittivity of free space.

A useful conclusion can be found from Eq. (2.9) is that the loss tangent is inversely proportional to the skin depth. In practice, that would mean microwaves can penetrate the materials thoroughly, with a low loss tangent, therefore, heating up the materials evenly.

Once the microwaves have successfully penetrated the materials, it would drag the dipoles to align with the field. When the dipoles rotate, they have to rotate against the friction between molecules, hence generating heat. Meredith (1998, p. 22) derived the equations to calculate the energy absorbed by the dielectrics. He firstly treats the dielectrics as a parallel-plate capacitor. The alternating current flowing through a capacitor can be calculated by the equation:

$$I = jV\omega C \quad \text{(A)} \qquad (2.10)$$

And the capacitance of the capacitor can be calculated by the equation:

$$C = A\frac{\varepsilon_0 \varepsilon_r}{S} \quad (F) \qquad (2.11)$$

Where A is area of the plate and S is the distance between the plates. As mentioned previously dielectric loss angle $\delta = \tan^{-1}\frac{\varepsilon''}{\varepsilon'}$ and $\varepsilon_r = \varepsilon' - j\,\varepsilon''$, substitute them with Eq. (2.11) into Eq. (2.10), that would yield:

$$I = V\,\omega\,\frac{\varepsilon_0 A}{d}\,(j\varepsilon' + \varepsilon'') \qquad (2.12)$$

The power dissipated by the material could be calculated by

$$P = V^*\text{real part of } I = V^2\omega\,\frac{\varepsilon_0 A}{d}\,\varepsilon'' \qquad (2.13)$$

Where ε_0 is the permittivity of the materials in free space and

$\varepsilon_o = 8.854187 \times 10^{-12}\ \text{Fm}^{-1}$.

In addition, the voltage stress in the dielectric is $E_i = \frac{V}{d}$ and also $\omega = 2\pi f$, substituting them into Eq. (2.13), that would give:

$$P = E_i^2\, d2\pi f\, \varepsilon_o\, \varepsilon'' A \qquad (2.14)$$

From Eq. (2.14), the power dissipation density p can be deduced as

$$p = \frac{P}{Ad} = 2\pi f \varepsilon_o \varepsilon'' E_i^2 \quad (2.15)$$

Eq. (2.15) shows the energy which aborted by the objects is proportional to dielectric loss and the electric field strength.

As the dielectric properties have been fully explained in the previous section, it is the time to analyse some materials' dielectric properties in relation to the heating of materials. Materials have high value of loss angle δ and loss factor ε'' tend to be heat up more efficiently. Table 2.2 indicates distilled water is the substance which suits the high value of loss angle and loss factor profile. On the other hand, water also reflects a significant amount of microwave since it has high value of ε'. It is not a matter as the metal wall of microwave oven will also reflect the microwave back to water, eventually most of the energy will be absorbed by the water.

Table 2.2: Dielectric properties of foods and other materials of 2.45 GHz at 20 °C (Platts 1991, p.5)

Material	Relative dielectric constant ε′	Loss tangent tan ε″
Distilled water	78	0.16
Raw beef	49	0.33
Mashed potato	65	0.34
Cooked ham	45	0.56
Peas	63	0.25
Ceramic (aluminium)	8–11	0.0001–0.001
Most plastics	2–4.5	0.001–0.02
Some glasses (Pyrex)	Approx. 4.0	0.001–0.005
Papers	3–4	0.05–0.1
Woods	1.2–5	0.01–0.1
Ice	3.2–3.3	0.0007–0.001

Compounds having a low molecular polarity would suffer less dielectric loss, in other words, generates less heat. For example, the compound like polystyrene, it has the loss factor ε'' of 0.0008 at 25°C and 2.5 GHz as shown in Table 2.3. Since it has extremely low value of dielectric loss factor, so it just wasting the electricity by trying to heat it up in microwave oven.

Table 2.4 shows epoxy has the value of loss tangent of 0.015 and the dielectric constant ε' of 3 at 25°C and 1 GHz. The loss factor ε" can be calculated with equation $\tan\delta = \frac{\varepsilon''}{\varepsilon'}$, thus the value of loss factor ε" is 0.045. Since epoxy has moderate value of loss tangent and loss factor, it is suitable for microwave heating.

Table 2.3: Dielectric properties of some materials (Meredith 1998, p.29)

		ε′/ε″		
Material	Temperature (°C)	30 MHz	1 GHz	2.5 GHz
Soda–silica glass, 20% Na_2O, 80% SiO_2	+25	6.2/0.1	5.8/0.09	5.7/0.09
Soil, loamy, 0% MC dwb	+25	2.48/0.03	2.46/0.008	2.44/0.004
Soil, loamy, 14% MC dwb	+25	17/10	20/2.5	20/2.5
Soil, sandy, 0% MC dwb	+25	2.55/0.033	2.55/0.012	2.55/0.007
Soil, sandy, 17% MC dwb	+25	20/30	20/0.3	17/0.3
c Organic solids				
Bakelite	+24	4.6/0.34	3.8/0.26	3.7/0.23
Butyl rubber (<98% isobutylene)	+25	2.35/<0.002	2.35/<0.002	2.35/<0.002
Ethyl cellulose	+25	2.9/0.045	2.8/0.048	2.7/0.051
Methyl cellulose	+22	4.6/0.46	3.7/0.26	3.4/0.20
Nylon 66	+25	3.2/0.072	3.08/0.049	3.02/0.041
Polycarbonate thermoplastic	+15	—	—	2.72/0.0034 at 9 GHz
Polypropylene				
Polystyrene	+25	2.56/<0.003	2.55/0.0008	2.55/0.0008
Polystyrene	+80	2.54/<0.0008	2.54/0.0009	2.54/0.001
Polyethylene	+25	2.25/<0.0004	2.25/0.0005	2.25/0.0007
Polyvinyl chloride (PVC)	+20	2.86/0.029	2.85/0.016	2.85/0.016
Polyvinyl chloride (PVC)	+47	3.0/0.043	2.9/0.025	2.8/0.021

Table 2.4: Dielectric properties of selected materials (NCS 1994, p.34)

Material	Frequency (GHz)	ϵ'	tan δ	Temp. (C)	Reference
Raw Beef	3.0	48.3	0.28	20	(Thuery, 1992)
Frozen Beef	2.45	4.4	0.12	-20	
Potato (78% water)	3.0	8.1	0.38	25	
Al_2O_3	3.6-3.8	9.02	0.00076	25	(Westphal and Iglesiad, 1971)
		9.69	0.00128	500	
		10.00	0.00930	700	
BN	8.52	4.37	0.00300	25	
Si_3N_4	8.52	5.54	0.00360	25	
Polyester	8.5	3.12	0.0028	25	(Westphal and Iglesias, 1971)
PTFE (Teflon)	2.43	2.02	0.00042	25	(Andrade et al., 1992)
PEI (Ultem)	1.0	3.05	0.003	25	(Bur, 1985)
Epoxy	1.0	3	0.015	25	(Bur, 1985)
Concrete (dry)	1.0	6.57	0.530	25	(Westphal and Iglesias,
Concrete (wet)	1.0	13.2	0.485	25	1971)

2.9.3 Thermal runaway

Meredith(1998, p.38) describes thermal runaway is the situation that occurs when the temperature rises quickly in some parts of the materials and the heat transfer rates to the surrounding parts are much slower than the rising temperature. Hence, the heat accumulates on that part of the material and may cause the material to decompose. Thermal runaway is often observed when heating up inhomogeneous mixture, in other words, the materials have a non-uniform density. Since the dielectric loss factor is proportional to temperature, if thermal runaway happened in the material, the hot spots tend to absorb more energy from microwaves, than other parts. Therefore the hot spots would get hotter and hotter, and that would accelerate the decomposition rate of the material. In addition, a simulation study of microwave heating (NCS 1994, p.37) suggested that if the power was injected to the materials to be less than a critical power level the thermal runway could be avoided. The results of the simulation have been displayed in Figure 2.5. Figure 2.5 shows when the electric field strength in microwave ovens exceed the critical value, the temperature of the material would suddenly rise to a much higher level.

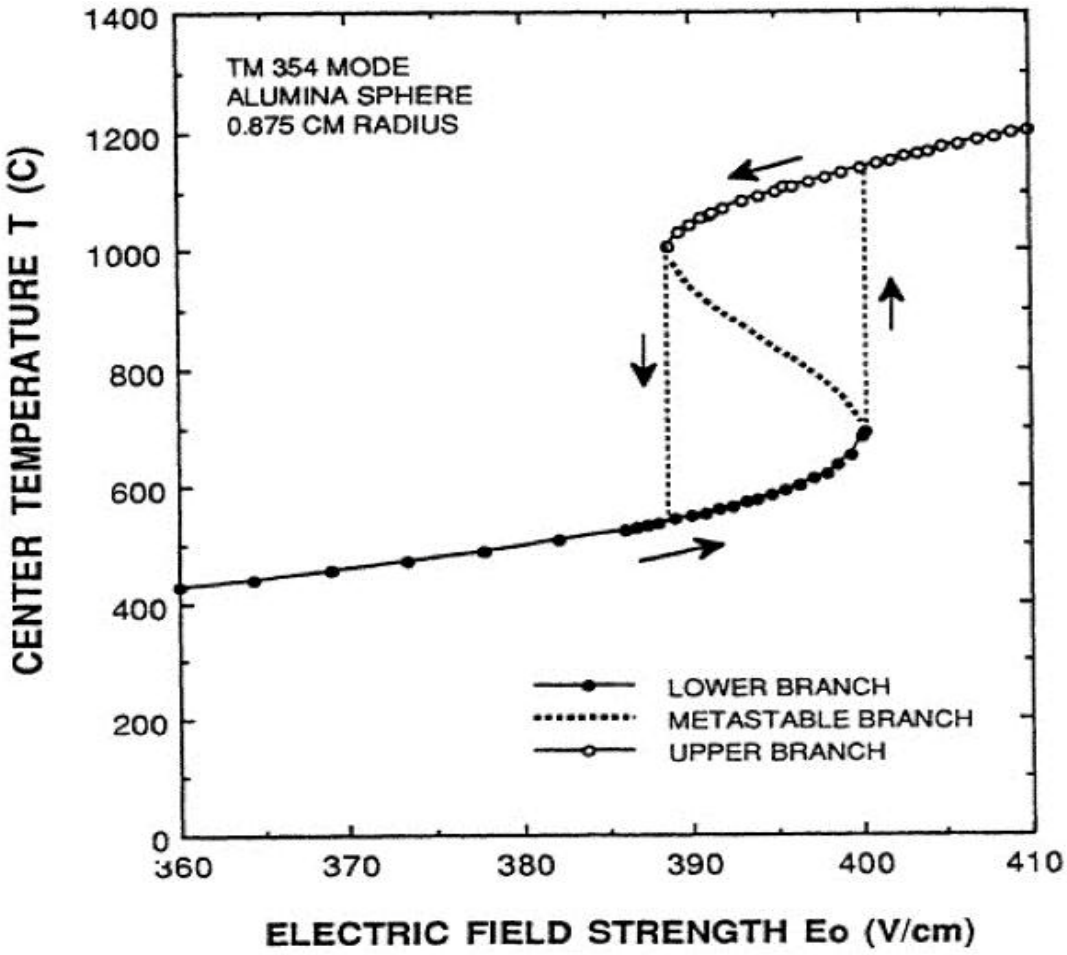


Figure 2.5: Predicted power- temperature response curve for an alumina sphere (NRC 1994, p.37)

Platts (1991) carried out a computer simulation to demonstrate the effect of the positioning of the heating object on the pattern of electric field by placing a potato in different position inside the microwave oven. The results are displayed in Figures 2.6 and 2.7. The results show that there are significant changes in the pattern of the electrical field after changing the position of the potato in the oven. Hence, to make sure the object is heated evenly, it should be placed in the center of the oven.

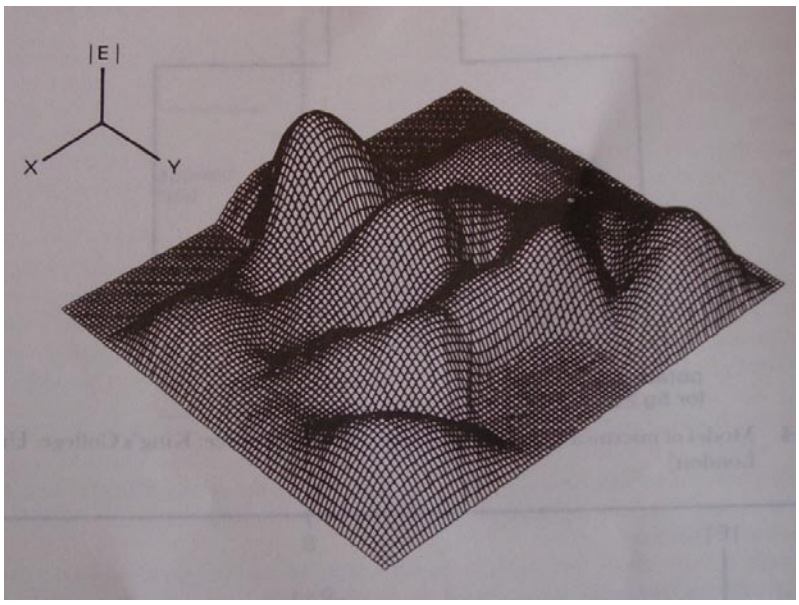


Figure 2.6: The field inside an oven with a potato at the centre of one side (Platts 1991, p.9)

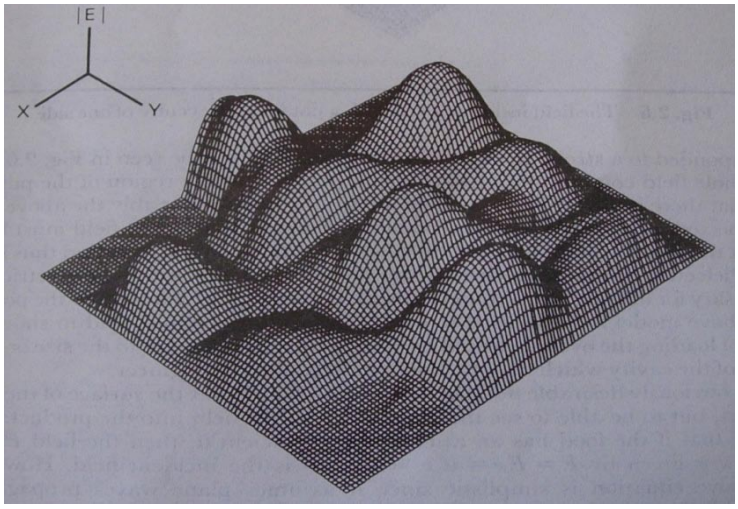


Figure 2.7: The field inside a microwave oven with a potato at one corner (Platts 1991, p.9)

2.9.4 Variable Frequency Microwave

As shown in Figures 2.6 and 2.7, the single frequency microwave always establishes the electric field with a fixed pattern. The area of the material impacted by a higher electric field strength would sustain more heat, hence that area would face a higher risk of thermal runaway. One way to overcome this problem is to apply the variable frequency

microwave to treat the material. With the variable frequency microwave, the electric field patterns would be frequently changed as the frequency of the microwave sweep through a range of frequencies.

2.9.5 Wall loss

Although the major portion of the microwave will be reflected back to the oven by the metal wall of the oven. A small amount of microwaves still can penetrate the metal wall, due to the fact of the slightly altered wavelengths.

2.10 Permittivity Measurement

Many methods and techniques have been developed in order to measure the dielectric constant and the dielectric loss of materials such as the dielectric probe and waveguide transmission and resonant cavity. Resonant cavity method is the most accurate method have been introduced by Ku et al. (1999) and verified by the experimental results. Ku et al.(1999) derived the formulas to calculate the dielectric constant ε' and the dielectric loss ε''. The equations are:

$$\frac{\omega_s - \omega_o}{\omega_o} = \frac{-(\varepsilon' - 1)t}{L} \tag{2.16}$$

$$\frac{1}{Q_s} - \frac{1}{Q_o} \cong \varepsilon''\left(\frac{2t}{L}\right) \tag{2.17}$$

Where ω_s = resonant frequency with sample;

ω_o = resonant frequency with empty cavity ;

Q_s = the Q factor for cavity with sample;

Q_o = the Q factor for empty cavity;

t = the thickness of the sample material in cm;

L = the length of Cavity in cm.

The desired resonant frequency with empty cavity can be controlled by the design of the dimension of the waveguide and cavity. The relationship between the resonant frequency and the dimension is described in Eq. (2.18):

$$(\frac{1}{2L})^2 + (\frac{1}{2a})^2 = (\frac{1}{\lambda_o}) \quad (2.18)$$

Where L = the length of the cavity in cm;

a = the width of the waveguide in cm;

λ_o = the wavelength of the electromagnetic field in free space in cm.

The value of ω_s and ω_o can be directly measured by the microwave network analyser. Therefore, the dielectric constant ε' can be easily calculated by using Eq. (2.16) for a piece of sample which its thickness is known. By definition the quality factor Q = $\frac{\omega \times energy\ stroed}{power\ dissipation}$, however, Q factor is not easy calculated by this equation. Hence, Ku et al.(1999) developed four methods to calculate Q factor. Here only introduce the ±90^o method. This method is established base on the equation Q = $\frac{f_o}{\Delta f} \times \frac{2\sqrt{|\rho min|}}{(1-\rho min)}$. Δf is the frequency differences between the frequencies correspond to phase angle of + 90^o and the frequency at phase angle of – 90^o. The ρ_{min} is the reflection coefficient correspond to the resonant frequency f_o. Figure 2.8 shows magnitude and phase angle of electrical field strength of microwave at different frequency measured by microwave network analyser.

Figure 2.9 shows the testing equipments connection configuration which was setup for the experiment carried out by Ku et al.(1999).

Due to resource constraints, although the method of resonant cavity can provide accurate measurement for the permittivity of the material, it will not be used in this project. In this project, instead to directly measure permittivity, the loss tangent tan δ of the composites will be measured. Loss tangent is important because it is proportional to the dissipated energy by the dielectric.

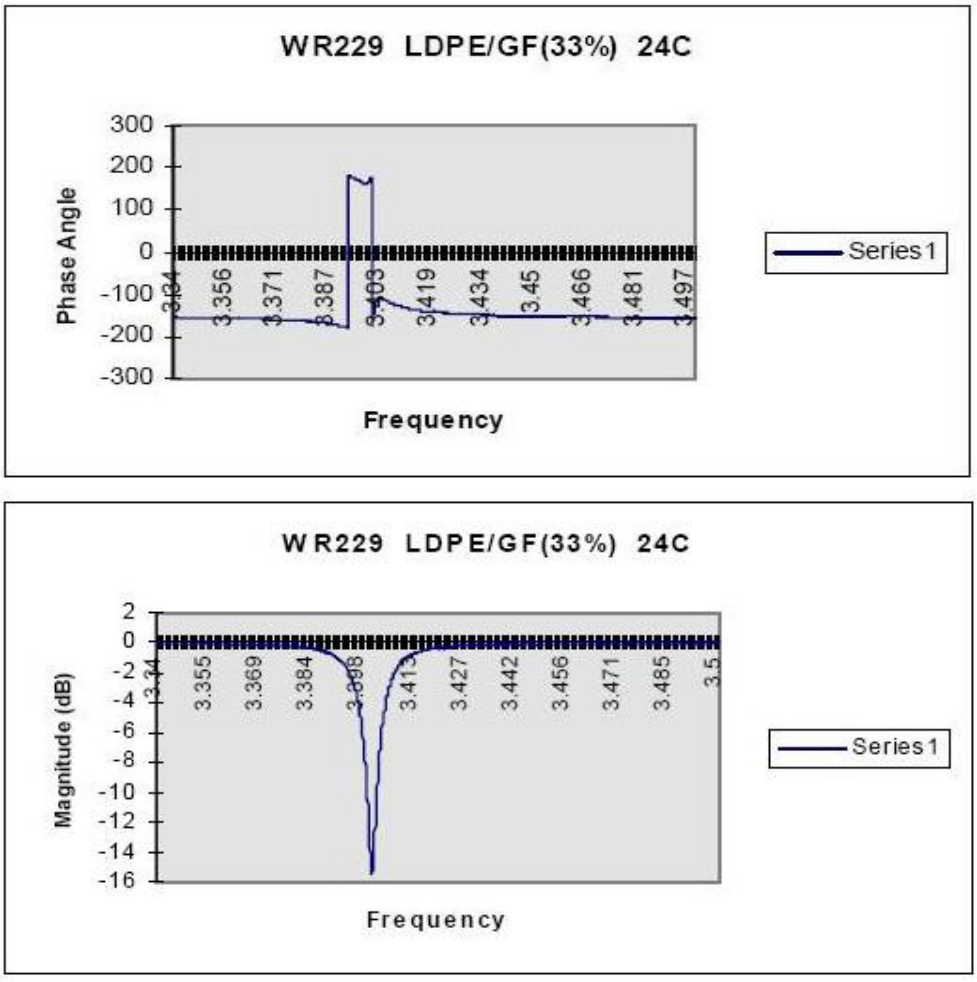


Figure 2.8: The phase and magnitude of the electric field at resonant frequency recorded by microwave network analyser (Ku et al.1999)

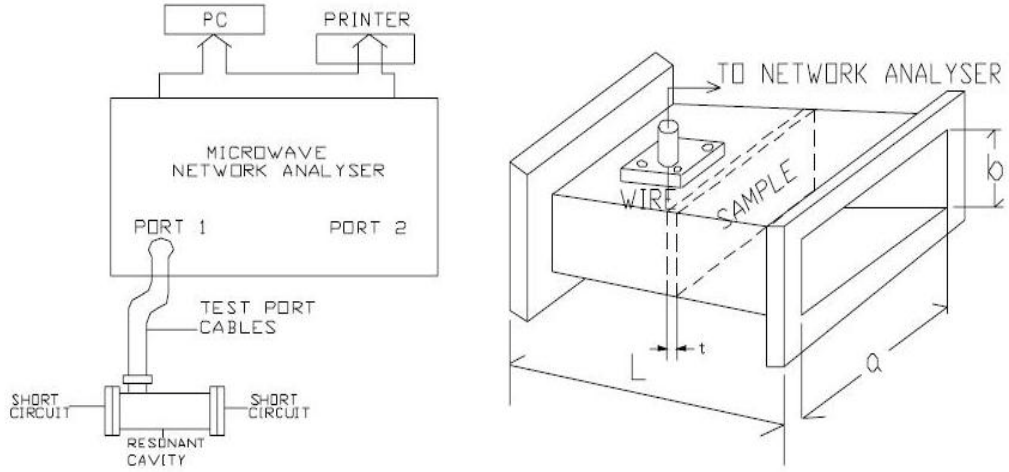


Figure 2.9: The Configuration of testing equipments for Cavity Method

2.11 Wave Guide

Inside microwave ovens, the microwaves are usually transmitted via waveguides. Figure 2.10 shows the path of microwaves travel inside the microwave oven. The microwaves are firstly generated by the magnetron tube, and then transmitted through the waveguide to the rotating metal fan. As the fan rotates, the microwaves are reflected at different angels. After the scattered microwaves hit the metal wall, they will bounce randomly inside the oven cavity until they absorbed by the heating object which is placed inside the oven.

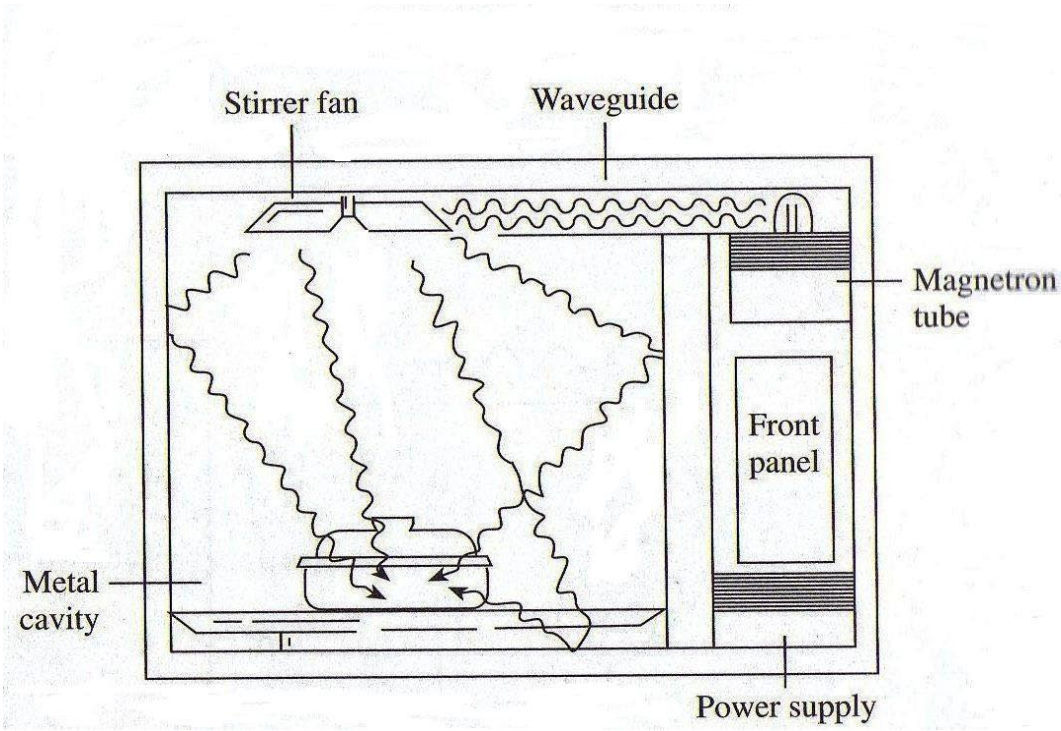


Figure 2.10: Microwave oven (Sadiku 2001, p.641)

2.12 Cut-off frequencies

Unlike transmission lines, microwaves can only start to propagate in the waveguide if he waves are above the cutoff frequency. In order to understand cutoff frequencies, the wave equation will be introduced here. The wave equation can be obtained by combining Eq. (2.4) and Eq. (2.5). That would yields:

$$\nabla \times \nabla \times E = -j\omega\mu\,(\sigma + j\omega\varepsilon)\,E \qquad (2.19)$$

It has been proved that for any vector, the following equation is valid:

$$\nabla \times \nabla \times E = \nabla\nabla \cdot \mathrm{E} - \nabla^2 E \qquad (2.20)$$

Moreover, if the medium is electrically neutral, by obeying Gauss's law, it can deduce that:

$$\nabla \cdot E = 0 \tag{2.21}$$

By substitution Eq. (2.19) and Eq. (2.21) into Eq. (2.20), Eq. (2.20) can be simplified to:

$$\nabla^2 E = j\omega\mu\,(\sigma + j\omega\varepsilon)\,E \tag{2.22}$$

Assuming the waveguide is filled with a prefect dielectric and perfect conducting waveguide walls. As the conductivity σ is equal to zero for the prefect dielectric, Eq. (2.22) can be further simplified to:

$$\nabla^2 E + \omega^2\mu\varepsilon E = 0 \tag{2.23}$$

Figure 2.11 shows a rectangular waveguide which has width of A and height of B. Since the waveguide walls have perfect conductivity, the electric field established inside the waveguide must satisfy the boundary condition, which is the electric field parallel to the surface of a perfect conductor is zero at the surface. Hence the general form of the field can be expressed as:

$$E(xyz) = \left[\sin\left(\frac{m\pi x}{a}\right)\right]\left[\sin\left(\frac{n\pi y}{b}\right)\right]e^{-\gamma z} \tag{2.24}$$

Or

$$E(xyz) = \left[\cos\left(\frac{m\pi x}{a}\right)\right]\left[\cos\left(\frac{n\pi y}{b}\right)\right]e^{-\gamma z} \quad (2.25)$$

Where a = the width of the waveguide in m

b = the height of the waveguide in m.

The equation for propagation coefficient γ can be found by substituting Eq. (2.24) or Eq. (2.25) into Eq. (2.23) that would yield:

$$\gamma^2 = \left[\left(\frac{m\pi^2}{a}\right) + \left(\frac{n\pi}{b}\right)^2\right] - \omega^2\mu\varepsilon \quad (2.26)$$

Moreover, by definition:

$$\gamma = \alpha + j\beta$$

Where α = the attenuation coefficient in Nepers/m;

β = the phase coefficient in radians/m.

The attenuation coefficient α describes the extent to which the magnitude of the wave is reduced with the distance in the direction of propagation from the source. The phase coefficient β measures the phase delay in radians per meter as the waves are transmitted down the propagation path of wave.

If the attenuation coefficient α equals to zero, there will be no wave attenuation occurring in the waveguide. This is the critical frequency which allows the wave to be launched by the waveguide without attenuation. If the frequency of the wave is lower than the cut-

off frequency, the wave cannot be launched through the waveguide. Thus, this frequency is called the cut-off frequency. The equation for cut-off frequency can be obtained by letting γ equals to 0 and substituting it into Eq. (2.26):

$$\omega^2\mu\varepsilon = \left[\left(\frac{m\pi}{a}\right)^2 + \left(\frac{n\pi}{b}\right)^2\right] \quad (2.27)$$

As ω = 2πf where f is the frequency of the wave, Eq. (2.27) becomes:

$$f = \frac{1}{\sqrt{\mu\varepsilon}} \times \sqrt{\left(\frac{m}{2a}\right)^2 + \left(\frac{n}{2b}\right)^2} \quad (2.28)$$

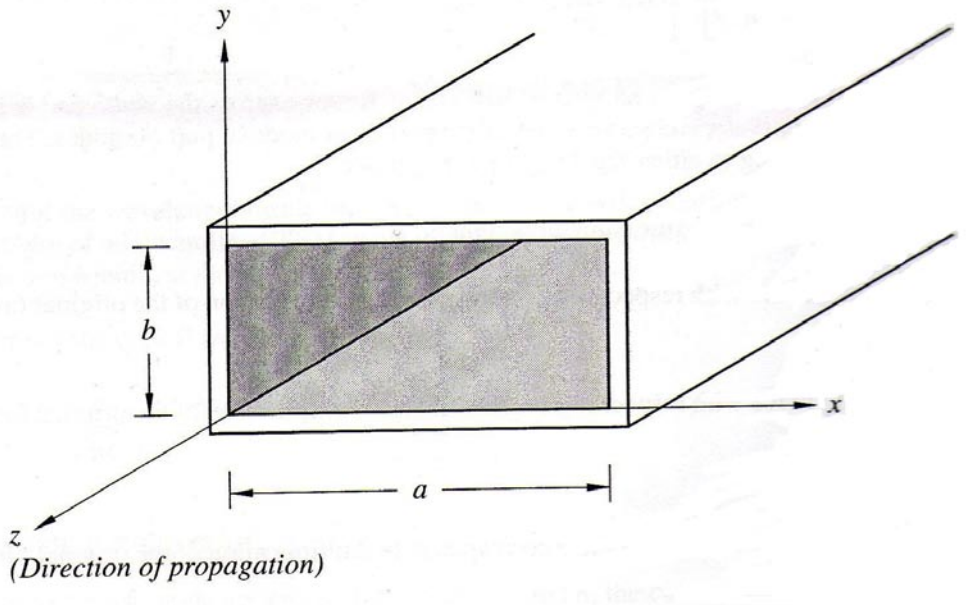


Figure 2.11: Coordinate system for rectangular waveguide (USQ 2009, p.13.1)

Figure 2.12 shows the cut-off frequencies for the different wave transmission modes. There are three main types of transmission waves, they are Transverse Electromagnetic waves (TEM), Transverse Magnetic waves (TM) and Transverse Electric waves. For

TM waves, only the electric waves are transmitted in the direction of propagation, therefore TM waves also called E waves as E is the electric field strength. Similarly, TE waves also can be called H waves as only the magnetic waves are transmitted in the direction of propagation. The subscripts of TE_{mn} which are m and n indicate the number of half sine waves across the width and the height of the waveguide respectively. The mode with the lowest cut-off frequency which is TE_{10} is called the dominant mode. In practice, the frequencies which are close to the cut-off frequency are avoided to be used. That is because of the great attenuation caused by the conductivity imperfection of the metal wall of the waveguide.

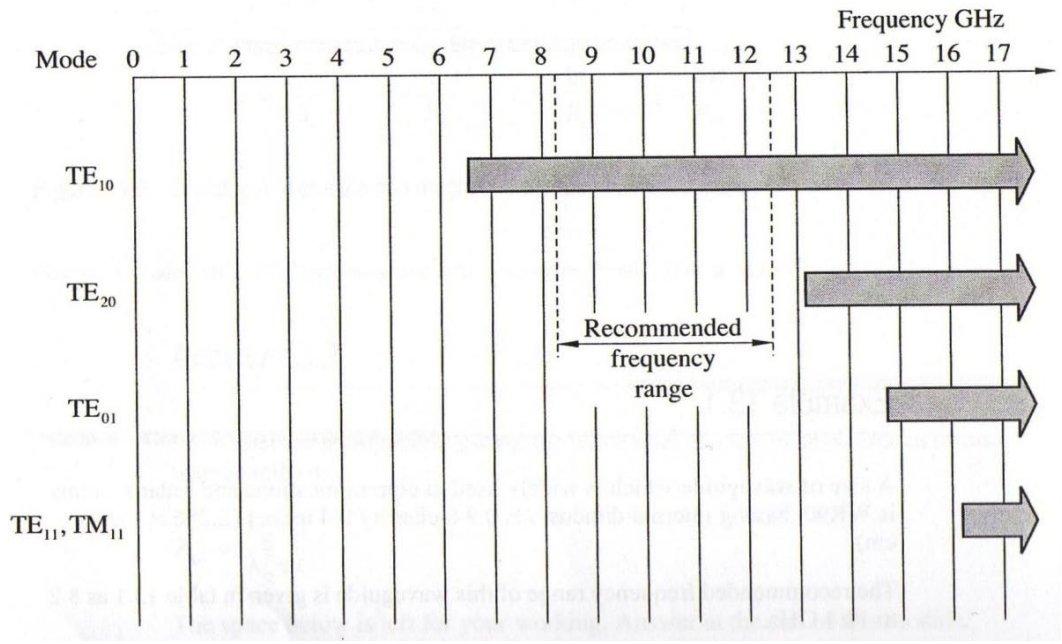


Figure 2.12 Cut-off frequencies for different transmission modes (USQ, p.13.14)

2.13 Work of others

There were some similar researches have been carried out by other people. One of the researches was to analyze the thermal properties of the sawdust reinforced vinyl ester composites post-cured in microwaves. This study was carried out by Dr Harry Ku, Dr Francisco Cardona and Mr Mohan Trada in 2010. The study found that the microwave cured and sawdust reinforced vinyl ester composites have higher glass transition

temperature than the samples cured in the thermal oven (Ku et al, 2010a). Hence, the microwave cured samples can withstand higher operating temperature than the thermal oven cured samples. The study also found the stiffness of the samples decreased with increasing percentage of sawdust.

Another research was to evaluate the thermal properties of calcium carbonate powder reinforced vinyl ester composites. This study was also carried out by Dr Harry Ku, Dr Francisco Cardona and Mr Mohan Trada in 2010. The study found that the thermal oven cured and calcium carbonate powder reinforced vinyl ester composites have higher glass transition temperature than the naturally cured samples (Ku et al, 2010b).

3. METHODOLOGY

3.1 Experimental equipment specifications

3.1.1 Microwave oven

The manufacturer of the microwave oven is Sanyo Electric Ltd. The model number of the oven is EM-S1563. The input voltage rating is 230 – 240 V at 50 Hz. The rated input current and power are 5.2A and 1.2kW respectively. The frequency of the microwaves generated by the oven is at 2450 MHz. The rated output power is 800 w (10 power levels).

3.1.2 Infra red handheld thermometer

The infra red handheld thermometer is made by Oakton Ltd. The model number of the thermometer is TempTestr IR WD-35625-10. The thermometer has the effective temperature measuring range from - 18°C to 260°C. The thermometer uses a 9 V battery. Its response time is 500 ms. Furthermore, the thermometer has the repeatability of ±2% of reading or ±2°C (±3°F).

The thermometer also has the accuracy as specified in the following:

- 25 to 260°C (77 to 500°F): ±2% or ±2°C (±3°F) whichever is greater;
- -1 to 25°C (30 to 77°F): ±3°C (±5°F);
- -18 to -1°C (0 to 30°F): ±4°C (±7°F)

3.1.3 Tensile test machine Test machine

The manufacturer of the universal test machine is MTS Ltd. The machine's maximum force generation capacity is 100 kN. The maximum ram travel distance is 150 mm. The operating software installed in the machine is TestStar IIS.

3.1.4 Loss tangent measuring devices

The instrument used for loss tangent measurement is the Agilent 4263B LCR meter. The input voltage rating is 240 V and the power rating is 45 VA.

3.2 Sawdust sifting

The required sizes of sawdust for this project are 300 μm, 425 μm and 1180 μm. To obtain the required sizes of sawdust, three corresponding sifters are used. To push the correct size of sawdust down into the container below the sifter, a piece of cloth was used to stir the sawdust inside the sifter. The sifter covered the container completely to prevent the unwanted size of sawdust drop into the container accidentally. Sawdust sifting was the most time consuming procedure for this experiment, especially for sawdust of 300 μm, it could take three or four days to obtain 200 g of 300 μm sawdust. Figure 3.1 shows the sifter of 425 μm.

Figure 3.1: 425 μm sifter used for sifting sawdust

Water will not dissolve in resin, hence the adhesion between the sawdust and resin would be significantly weakened if water is present in the mixture. Therefore, before the sawdust can mix with the resin, the moisture must be removed from the sawdust. Therefore it has to be placed in the oven and heated to 90°C for a couple of hours.

3.3 Moulds preparation

Figure 3.2 shows the mould used for preparing the samples of tensile test. Three of these moulds are made of a sheet of aluminum and purchased from BigW supermarket. They have strong bottom surfaces which are tough enough to support the weight of the specimens without any distortion. The moulds are 280 mm long, 210mm wide and 15mm deep. These moulds were specifically chosen to make type 2 tensile test specimens as required in AS 1145.4 (2001). The moulds were then completely covered by wax paper before the mixtures of the specimens were poured in the moulds to prevent the specimens from sticking to the surface of the mould. This is a very important step, if strong force is used to separate the specimens that might cause visible or non visible cracks in the samples, which would significantly change the tensile test results.

Figure 3.2: The mould for casting tensile test samples

Figure 3.3 shows the mould used to casting the samples for loss tangent test. This mould was consisted by two PVC plates. A piece of wax paper was placed between the upper and lower plates to prevent the specimens from adhering to the surfaces of the plates. As illustrated in Figure 3.3, there were two figure size slots. It was very difficult to fold the wax paper to cover such a small area, hence a small amount of wax was smeared on the surface of the slots. The wax also was used to seal the edges of the slots to prevent the mixture leaking through the gaps between two plates. Screws were also used to hold the two plates together.

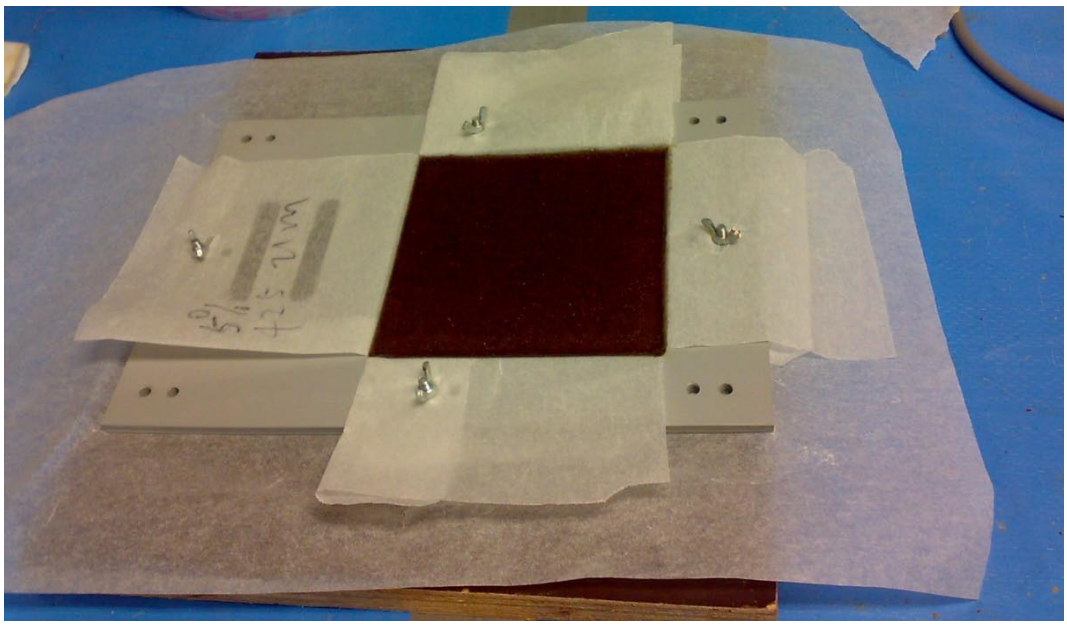


Figure 3.3: The mould used for casting the samples for loss tangent measurement

3.4 Mixing the samples

The resin, hardener and sawdust are carefully measured on an electronic scale separately before pouring into one container to mix together. Care needed to be taken when pouring the resin from the weight container into the mixing container. The resin is a very sticky substance, and a considerable amount of residual resin can stick to the surface of the weight container after pouring it into the mixing container. It is necessary to move the residual resin into the mixing container with the assistance of a spoon to make an accurate sample ratio. After the resin, hardener and the sawdust were poured into the mixing container, they were stirred for 15 minutes. The well mixed samples were then poured into the moulds for curing.

3.5 Specimens cured in room temperature

Figure 3.4 shows the specimen is placed on a flat surface to cure for more than 24 hours before post-cured in microwave oven. The specimen is too soft to remove from the mould if the curing time is less than 24 hours. Each sample was marked with its

percentage of sawdust by weight and the size of the sawdust was used in the mixture in order to differentiate between the samples.

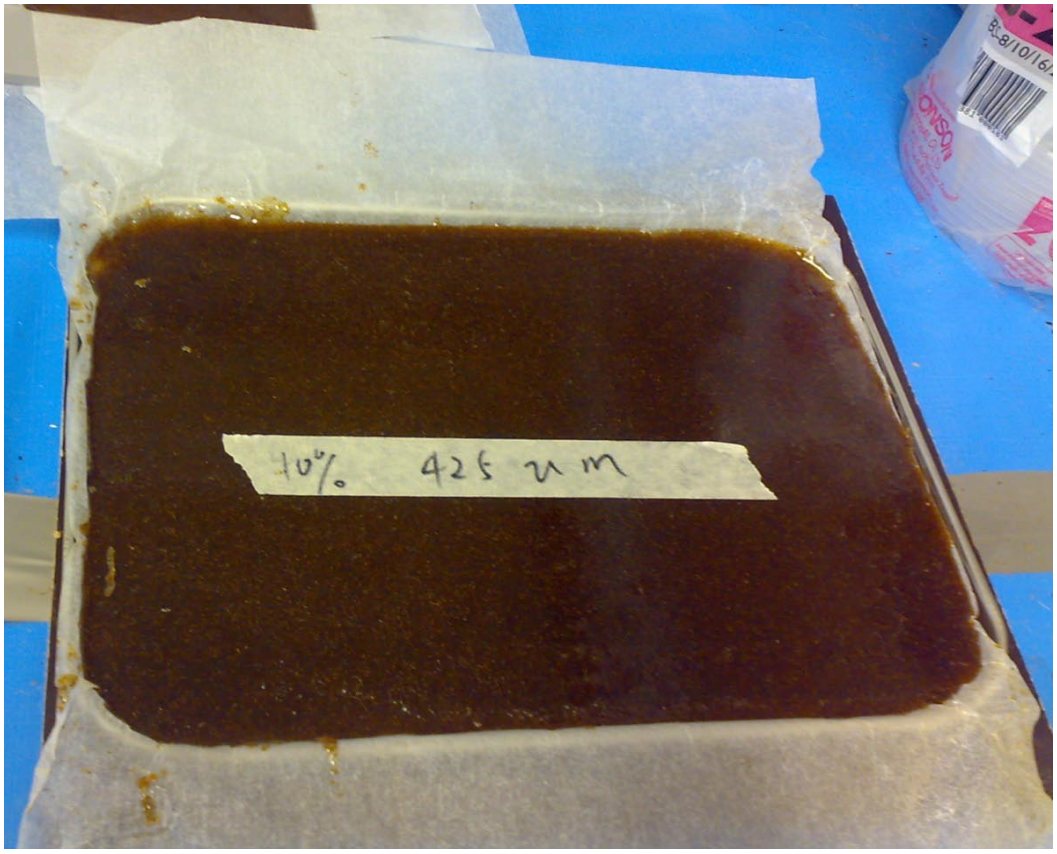


Figure 3.4: The specimen curing at room temperature

3.6 Dimension of Specimens

In order to produce standardised test results, the tensile test was executed under the guidance of AS 1145.4 (2001). The type 2 specimens which are rectangular bodies without end tabs were made for this project. AS 1145.4 required the dimension of type 2 specimens should be greater or equal to 250 mm in length, 25±0.5 in width and 2 to 10mm in thickness. Hence, after the specimens were post-cured in the microwave, they are cut into to 250 mm in length, 25 mm in width to satisfy with AS 1145.4 (2001). Figure 3.5 shows the samples which are cut into the required size.

Figure 3.5: The samples cut by saws

3.7 Post-cure in the microwave oven

After the specimens were cured in room temperature for more than 24 hours, they then were placed into the microwave for post–curing. Figure 3.6 shows the The fly-ash removal reinforced microwave oven used for this project. Before starting the oven, a glass of water had been placed into the microwave to absorb the excessive microwave energy and prevent overheating. The specimens were placed into the microwave to heat up to 40 ^{o}C. This process took 8 minutes with selected power level of 160 W to reach required temperature. The temperate of the specimens was measured by an infra red handheld thermometer. Figure 3.7 shows the infra red handheld thermometer used for this project. Some hot spots were observed when the thermometer was moved along the specimens. The observed temperature differences can be as significant as 20 ^{o}C. No thermal runaway occurred, in other words, there was no burned spot or material decomposition to be observed. Hence 160 W is an adequate power level to heat up sawdust reinforced epoxy composites. After the heated specimens cool down to room temperate, the specimens were then again placed into the oven and heated to 50 ^{o}C. It required 15

minutes with selected power level of 160 W for the specimens to reach 50 ^{o}C. The specimens then left in the oven to cool down to room temperate. The specimens were again heated to 60 ^{o}C. This time, it took 20 minutes with selected power level of 160 W for the specimens to reach 60 ^{o}C. One thing needs to be mentioned here, for safety and health reason, this microwave oven had been modified to remove the curing fly ash via an attached air duct. All the windows should keep open during the process to keep good air circulation in the room.

Figure 3.6: The fly-ash removal reinforced microwave oven

Figure 3.7: The infra red handheld thermometer

3.8 Post-cure in conventional thermal oven

The samples are post-cued in thermal oven at three stages. At the first stage, the samples are post-cured in oven for 16 hours at 40°C. After the first stage, the samples then post-cured for other 16 hours at 50°C, that is the second stage. After the second stage, the samples then heated up to 60°C for 8 hours. After all three stages finished, the samples are ready for testing.

3.9 Tensile test

The tensile test was carried out with the universal testing machine which shown in Figure 3.8. The gauge length adopted for this test was 150 mm and test rate was 2 mm/min. Before place the samples into the grips, the width and thickness were measured by an electronic vernier caliper and entered into the computer program to calculate the young's modulus and tensile strength. Figure 3.9 shows the caliper which is used for the dimension measurement of the entire project.

Figure 3.8: Universal Test machine

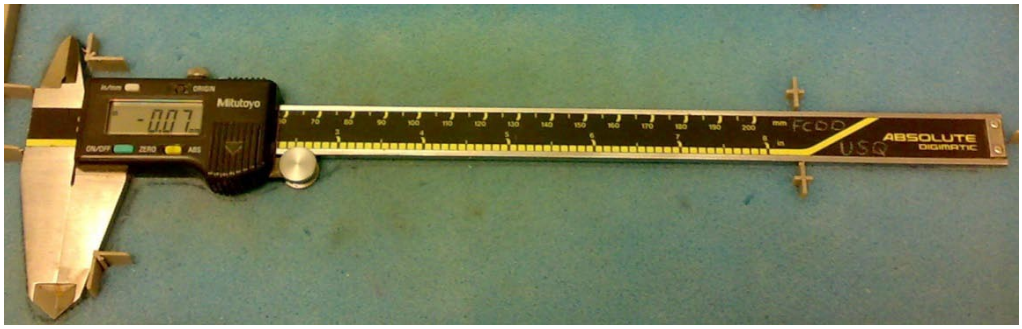


Figure 3.9: Electronic vernier caliper

The samples were clamped and held vertically by the jaws which were attached to the movable cross head of the machine. There are several sizes of jaws available. Inadequate jaw sizes could cause the sample to slip between the grips. Moreover, selecting the appropriate clamping pressure is also an important step before starting the test. If too much clamping pressure is applied, it may break the samples. Or if the pressure applied is not strong enough to hold the sample still, the sample may slip during the test. The universal testing machine is connected to a computer. All the test results were sent to the computer and displayed on the screen. In the normal case, the elongation of the test specimen should increase as load increases. If the slipping of the sample happened, the load to extension curve which is displayed on the screen would go flat. In this case, bigger jaws are required to replace the jaws of inadequate size. Since the testing machine is driven by hydraulic force, extra attention is needed to prevent the breaking of the sample from excessive clamping pressure. For personnel safety, the guarding shield should be closed before stating the test.

3.10 Dielectric constant and loss tangent measurement

The method used for this project was called the parallel plate measurement method**.** Unlike the resonant cavity method, this method is established based on some assumptions, hence this method can only provide approximate values for the loss tangents and permittivity. This experiment was carried out in a specially designed room. The wall of the room and the roof were made of metal and earthed. The earthed metal wall provided a shielding to protect the room from the outside electromagnetic interference. The metal door has to be closed before the experiment starts. Figure 3.10 shows the equipment set-up for the loss tangent and dielectric measurement. First, the test sample was placed between two copper plates. The copper plates are 110mm x 110 mm which are slightly smaller than the sample which is 120mm x 120mm. Then copper plates and the sample were inserted into two wooden clampers. The copper plates and the sample have to be bolted tightly together to minimize the air gap between the plates and the sample. The two measurement leads (black and red) of LCR meter were connected to the wires which were soldered to the middle surface of the two copper plates to allow the current to flow. After the LCR meter was turned on, the measuring parameters Cp and D which were parallel capacitance and dissipation factor

respectively were selected for the measurement. The dissipation factor D is also known as loss tangent. The parallel capacitances and dissipation factors were measured at 100 Hz, 1kHz, 10 kHz, 20 kHz and 100 kHz respectively. Ideally, the measurement should be conducted at higher frequencies since the frequency range of microwave is between 300 MHz to 1000 GHz. However, with the limitation of signal generating capability of the LCR meter, the maximum signal could be generated by the LCR meter is 100 kHz. Although the measurement was carried out at lower frequencies, the results could still give certain indications for the electrical properties of the test materials. The test results are read off the screen of the LCR meter and entered manually into a spread sheet for analysis.

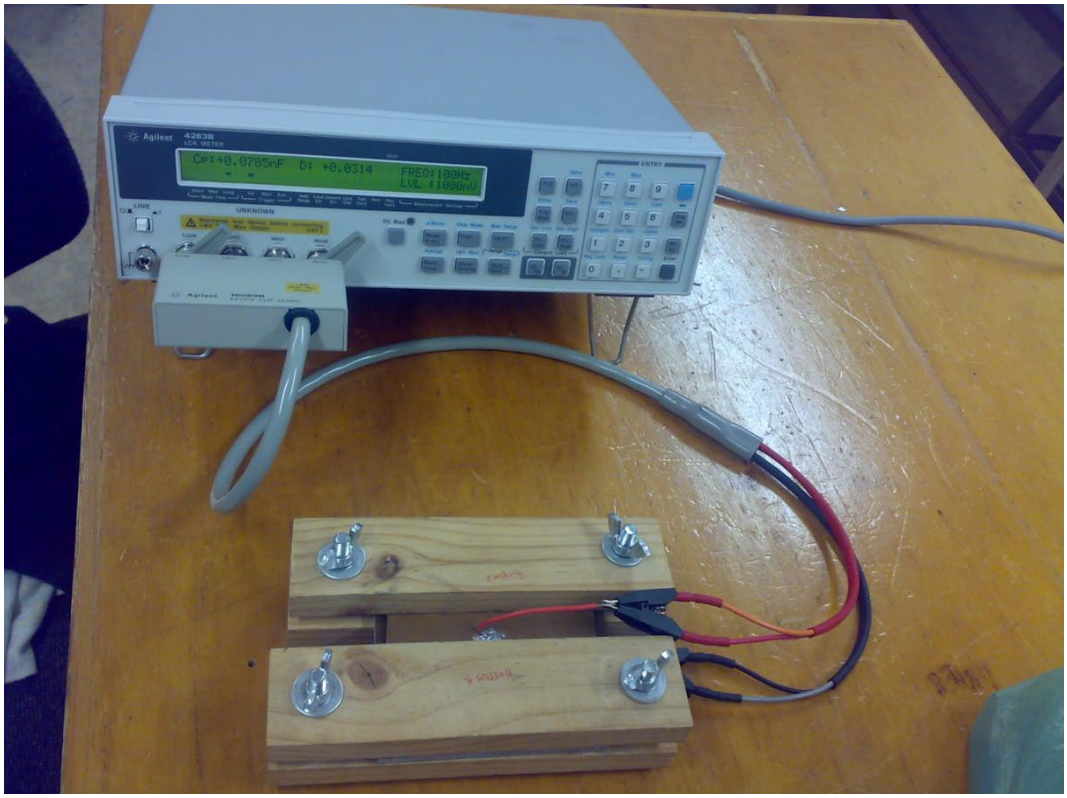


Figure 3.10 Loss tangent measurement equipment set-ups

Figure 3.11(a) shows the equivalent electrical circuit for the samples under test. The red lead of the LCR meter carried a small amount of current flow into the copper plate, and the black lead carried the current flow back to the LCR meter. The two copper plates with the test sample in the middle formed a parallel plate capacitor. Cp is the parallel

capacitance of the samples and G is the shunt conductance of the sample. By applying the ac voltage across two copper plates, the ac current will flow through the equivalent circuit. Figure 3.11(b) shows the phasor diagram for the currents flow through the equivalent circuit. The Phase difference between Ic which is the current flows through the capacitance and Ig which is the current flows through the conductance is 90o out of phase. The angle between the conductance current Ig and the resultant current I is the loss angle δ. Hence the loss tangent can be determined by measuring the phase angle of the resultant current respect to the capacitance current Ic. Therefore, the LCR meter is able to directly measure the loss tangent.

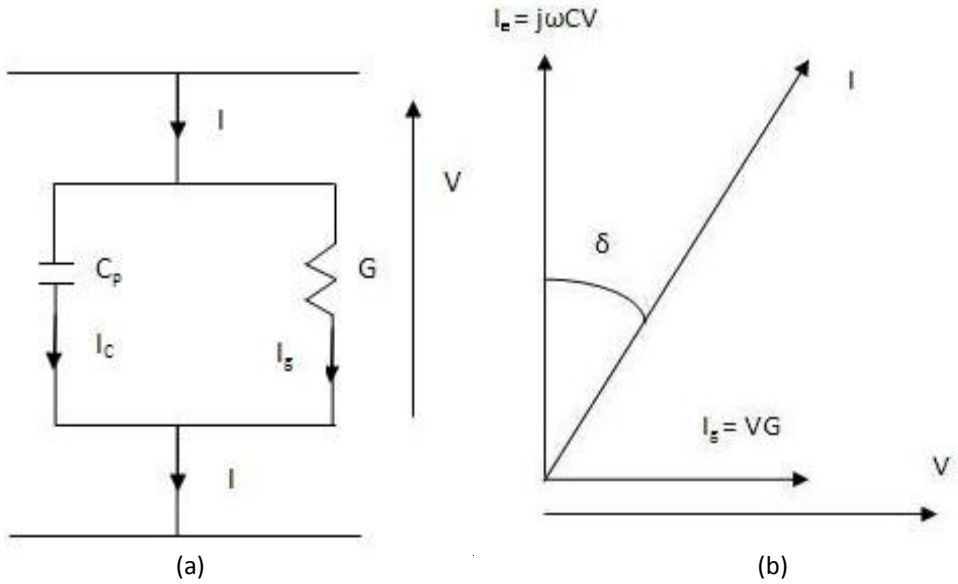


Figure 3.11: (a) equivalent circuit for the sample under test (b) phasor diagram

The LCR meter is also able to directly measure the capacitance and the conductance of the material. The permittivity can be calculated by rearranging

Eq (2.10):

$$\varepsilon_r = \frac{S \times C_p}{A \times \varepsilon_0} \qquad (3.1)$$

Where S = the average thickness of the sample in mm^2

A = the surface area of the plate in mm^2

$\varepsilon_o = 8.854187 \times 10^{-12}\ Fm^{-1}$

C_p = the measured parallel capacitance

Hence, once the area of surface of the plate, the thickness of the sample and the parallel capacitance are measured, the value of ε_r can be easily determined by Eq (3.1). Furthermore, the conductance of the samples is given by:

$$G = \frac{\sigma' A}{S} \quad (S/m) \tag{3.2}$$

Where σ' is the effective a.c. conductivity

and $\sigma' = \sigma + \omega\varepsilon_o\varepsilon''$

Where σ is the DC conductivity of the material

ω is the frequency of the test signal

Divide Eq. (2.10) by Eq. (3.2), it would yield:

$$\frac{C}{G} = \frac{\varepsilon_o\varepsilon'}{\sigma + \omega\varepsilon_o\varepsilon''} \tag{3.3}$$

Assuming the DC conductivity σ equal to zero for good dielectrics, Eq. (3.3) becomes:

$$\frac{C}{G} = \frac{\varepsilon'}{\omega\varepsilon''} \tag{3.4}$$

Substitute Eq. (2.7) into Eq. (3.4), the new equation becomes:

$$\tan\delta = \frac{G}{\omega C} \tag{3.5}$$

Hence, alternatively the loss tangent can be calculated by measuring the conductance and the capacitance of the material at different frequencies.

3.11 Possible measurement errors

The capacitances which measured by the parallel plate measurement method were greater than the actual value of the capacitance of the material due to the presence of the edge effects. In practice, the electric field is not uniformly distributed at the edge of the two metal plates. As shown in Figure 3.12, the electric flux line actually curves outwards beyond the edges of the plates. Hence the measured capacitance actually is the capacitance of the samples plus the stray capacitance at the edges of the plates.

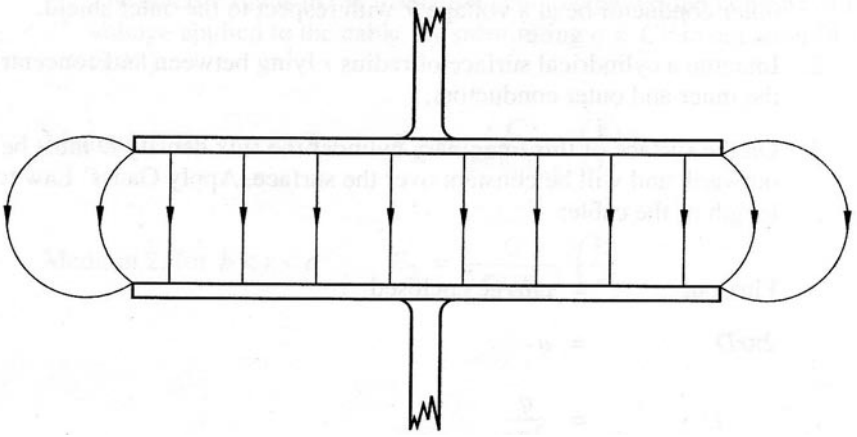


Figure 3.12: Edge effects in a parallel plate capacitor (USQ 2009, p.9.5)

Another possible measurement error could be caused by the air gaps between the plates and the samples. Figure 3.13 shows the airgap effects in the capacitance measurement. Since air is also a dielectric substance, it is able to hold the electric filed and contribute to the measured capacitance. As shown in the Figure 3.13, the equivalent model for the air gap effects will be the series connection of the capacitance of the sample and the capacitance of the air gap. The measured capacitance becomes:

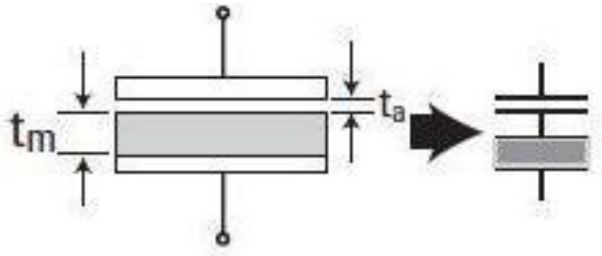


Figure 3.13: Airgap effects (Agilent 2008, p.6)

$$\frac{1}{C} = \frac{1}{C_p} + \frac{1}{C_a} \tag{3.5}$$

where

C_p = the parallel capacitance of the sample

C_a = the capacitance of the air gap

C = measured capacitance

It can be concluded from Eq. (3.5) that the more air gap exists between the sample and the copper plates, the smaller the measured capacitance would be. The samples should be placed as flat as possible when it is casted in the mould to minimize the air gap effects.

3.12 Dynamic Mechanical Analysis (DMA) test

DMA test is a technique to measure the visco-elastic properties of the materials which included storage modulus, loss modulus and phase angle tanδ. Clarification needs to be noted that the phase angle tanδ is different from the loss angle tanδ which is an electrical property. The main principle of the DMA test is an established fact based on the linearity between the stress and strain. The stress and the strain can be measured by applying a force to make the material oscillate.. There are two methods implemented to

apply the force. One of the methods is called free oscillation method. In this method, the force is only applied to the material for a very short period. Once the material starts oscillating, the external force is removed and allows the material to oscillate freely. Another method is called forced oscillation. In forced oscillation method, the oscillating force (usually sinusoidal oscillating force is used) is continuously applied to the material throughout the whole period of testing. Hence the material tested by the forced oscillation method would oscillate at the exactly same frequency as the applied force. Therefore, the forced oscillation can provide more reliable results than the free oscillation method when performing a temperature sweep. In this project the forced oscillation method is used and the test is conducted by applying a sinusoidal oscillating force to the material under test. Visco-elastic materials have two distinct physical states. Before reaching the glass transient temperature, the material is in an elastic solid state. The strain occurred to the material is proportional to the stress applied to the material and in phase, hence the material oscillates at the same frequency as the applied stress. After passing the glass transient temperature, the material starts turning into a viscous fluid state. In this state, the resulting strain gradually lags the stress as the temperature increases. When the material completely turns into the viscous fluid state, the strain lags the stress by 90 degree. The phase lag between the strain and the stress is called phase angel δ.

The storage modulus E' is the measure of the stiffness of the elastic material. It is proportional to the energy stored during the period of material elastic deformation occurs. Since the deformation is elastic, the process is reversible and the energy stored at this stage can be released back to the system. Thus the energy consumption at this stage is insignificant. However, after the testing temperature rises above the glass transition temperature, the material turns into the viscous fluid state. At this state, the resulting strain starts lagging behind the applied stress. As the strain and the stress are not in phase, the absorbed energy is converted to heat and cannot be recovered. The loss modulus E'' is the measure of this non-reversible heat loss. The loss factor tan δ is the ratio of loss modulus E'' to storage modulus E'. It is a measure of the energy lost, expressed in terms of the recoverable energy, and represents mechanical damping or internal friction in a viscoelastic system (Hanser 2006). Figure 3.14 shows the typical storage modulus and tan δ curves. At the beginning of the test, the storage modulus is at

its maximum value because the temperature is the lowest. As the material turns from elastic solid state to viscous fluids state after the testing temperature rise above the glass transients temperature, the storage modulus which representing elastic property drops dramatically to nearly zero MPa while the loss modulus reaches the maximum. Hence, maximum point on the curve of the loss modulus can be used to determine the glass transient temperature. There are two main test modes are used for DMA test. The mode was chosen for this project is temperature sweep. With this mode, the material under test is subject to a sinusoidal stress which is fixed at a low constant frequency while increasing the sample temperature. Another test mode is so called frequency sweep mode. In this mode, unlike the above mode, varying the temperature, the frequency of the sinusoidal stress is swept over a range of frequencies.

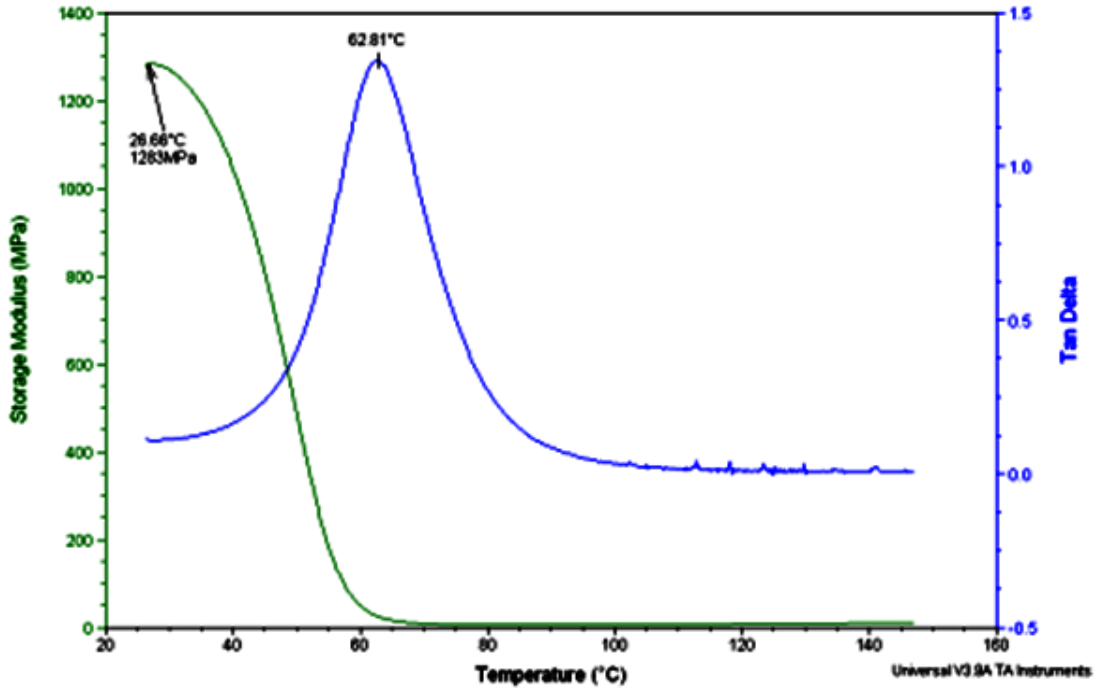


Figure 3.14: The DMA test results for naturally cured epoxy resins reinforced with 10% 425 µm sawdust

Figure 3.15 shows the DMA instrument used for this project. The sample is firstly placed in the clamps, and held tightly by the clamps. After the sample been place in the clamps the cover would move down and close the test chamber. That gives better control for the testing temperature in an enclosed environment. The stepper motor

underneath the clamps then drives the shaft to move the sample with it. The applied stress and resulting strain are sent to the computer for analyzing. The temperature ramp rate for this test was chosen at 3^{o}C per minute. The maximum test temperature was set at 270 ^{o}C. The sample dimension used in this test was 60mm long by 10mm wide by 6 mm thick.

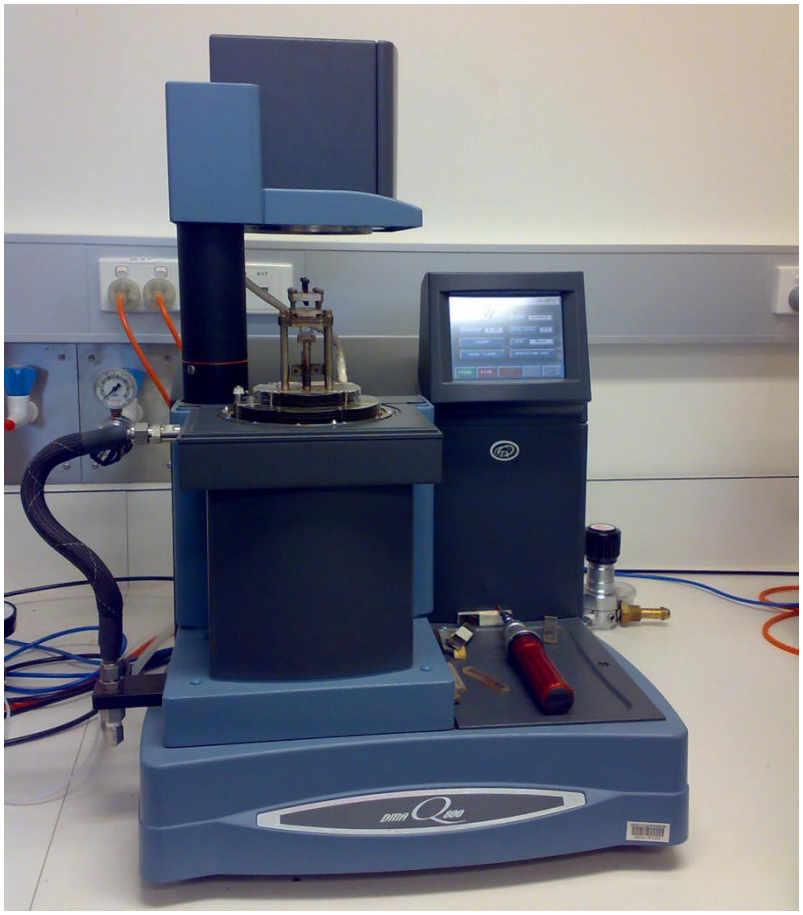


Figure 3.15: DMA instrument

3.13 Microscope Analysis

Figure 3.16 shows the optical microscope used in this proejct. It is an Olympus SZ-PT optical microscope. It has a digital camera attached on the top. The pictures captured by the digital camera are magnified 50 times before being sent to the computer.

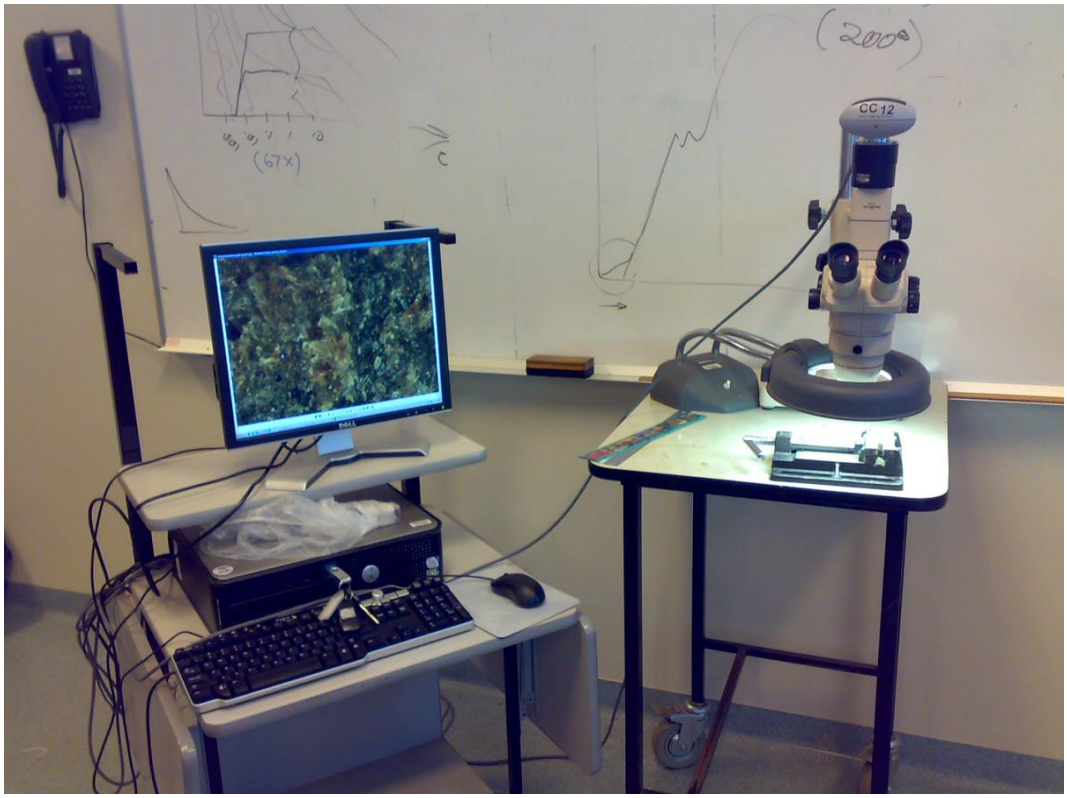


Figure 3.16: The optical microscope used in this project

4. RESULTS AND DISCUSSION

4.1 Loss tangent and Dielectric constant test results

According to USQ (2009, p11.9), the electrical classification of materials can be identified by measuring the loss tangent. If the loss tangent is greater than 100, the material is classified as conductor. On the other hand, if the loss tangent is less than 0.01, the material is classified as a dielectric which would stop the current to flow. The material fall in between the range is classified as a quasi-conductor. Loss tangent is proportion to the dissipation in the dielectric. In order to suit for high-speed electronic applications and higher soldering temperature, it requires to decrease the dielectric constant ε' and loss tangent and increase the glass-transition temperature Tg. High loss in transmission would result in reducing signal intensity and increasing thermal noise present(Morin, 2007). The dielectric constant of printed circuit boards (PCB) affects the signal speed of the circuit by the equation:

$$v = \frac{c}{\sqrt{\varepsilon_r}} \text{ m/s} \qquad (4.1)$$

Where C = 2.9979 25 × 10^8 m/s which is the speed of light.

4.1.1 The loss tangent measurement

Figure 4.1 shows the oven post-cured pure epoxy resins have higher value of loss tangent than the microwave post-cured pure epoxy resins, and the pure resin cured at room temperature. Since loss tangent is proportion to the heat dissipation in the dielectric, hence the oven post-cured pure epoxy resins are more effective to absorb the microwave energy. At 100 Hz, it has much higher value of loss tangent than the values at other frequencies. This can be explained by considering that the signal frequency is too slow to align the dipoles of the molecules with the change of the signal. Other the other hand, loss tangent is also inversely proportional to skin depth, therefore, the

distance which microwave can penetrate into the oven cured pure epoxy resins is shorter than the distance which the microwave can penetrate into the microwave cured pure epoxy resins. Higher value of skip depth means the samples can be heated by the microwave more uniformly.

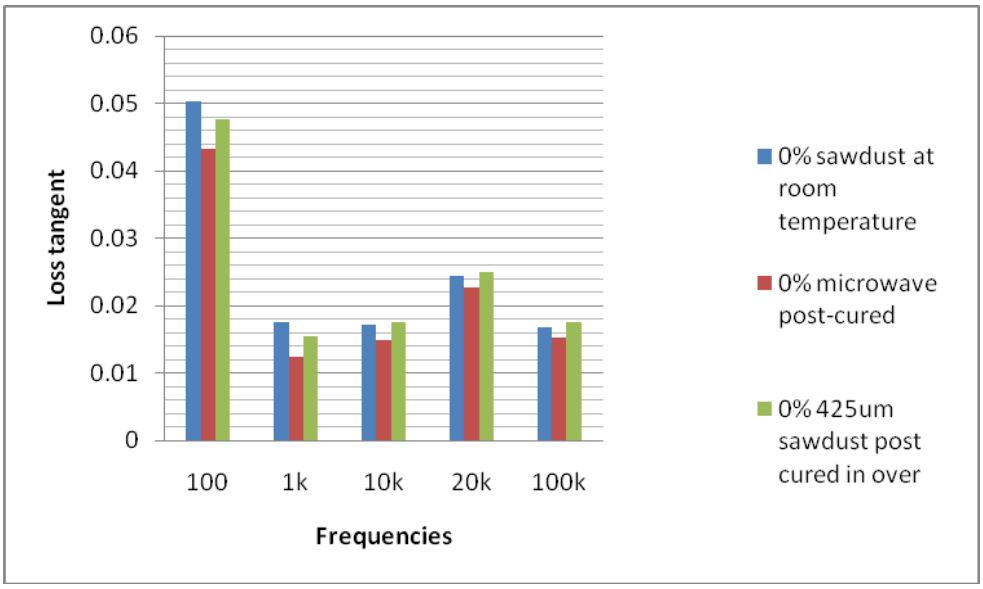


Figure 4.1: Comparison of loss tangent from different curing method of pure epoxy resins

Figures 4.2, 4.3 and 4.4 show the sawdust reinforced epoxy resins exhibit different dielectric behaviors with the pure epoxy resins. The microwave cured sawdust reinforced epoxy resins have higher values of the loss tangent than those oven cured samples and the sample cured at room temperature. The results obtain from sawdust reinforced epoxy resins are opposite with the results obtained with pure epoxy resin samples. As mentioned before, loss tangent is proportional to the heat dissipation in the dielectric, therefore, for sawdust reinforced epoxy resins, microwave cured samples are more efficient to absorb the microwave energy. However, as mentioned before that loss tangent is also inversely proportional to skin depth, therefore, the distance which microwave can penetrate into microwave cured sawdust reinforced epoxy resin is shorter than the distance which microwave can penetrate into oven cured sawdust reinforced epoxy resin.

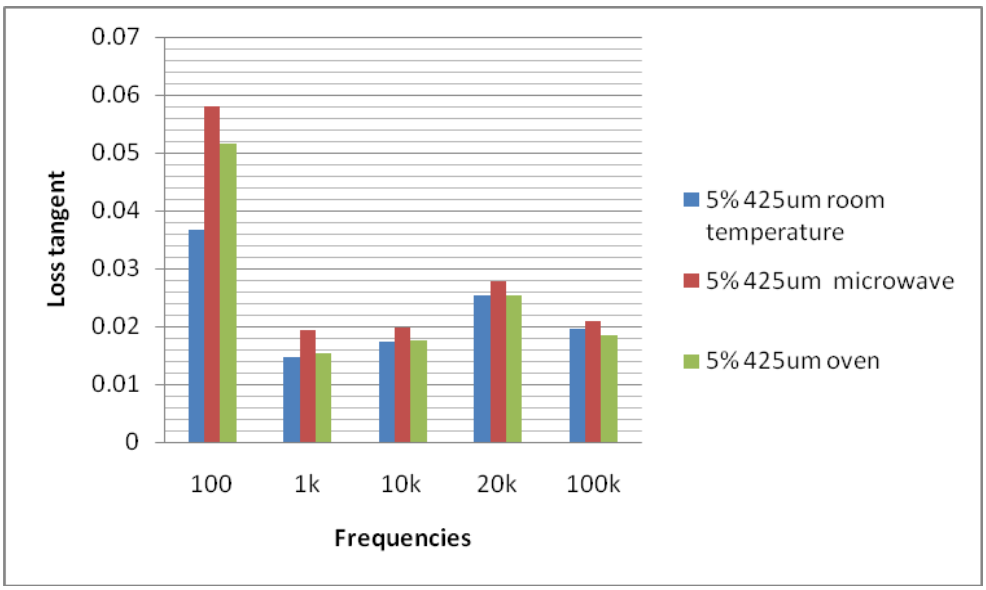


Figure 4.2: Comparison of loss tangent from different curing method of epoxy resins reinforced with 5% 425 µm sawdust

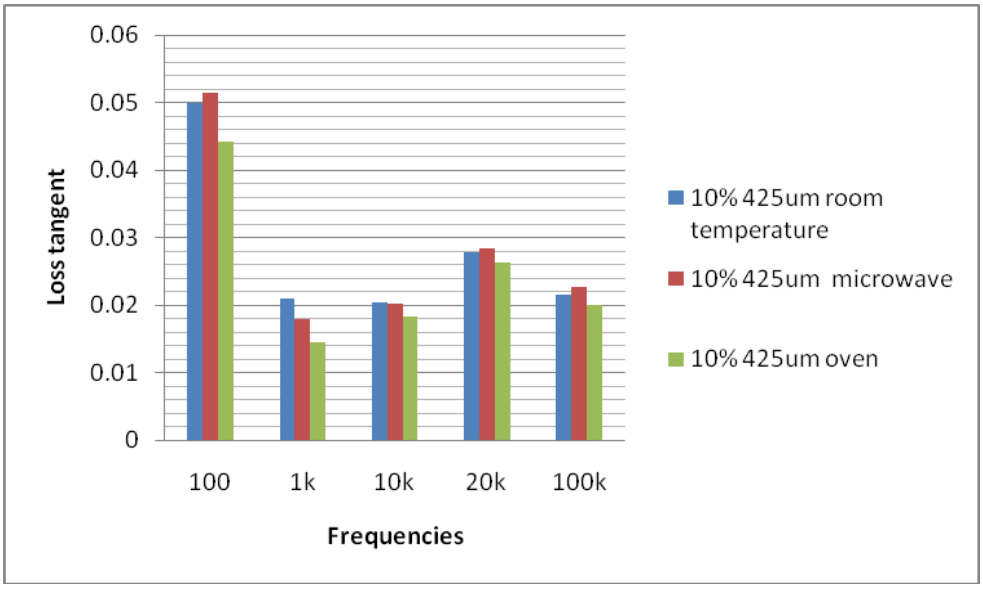


Figure 4.3: Comparison of loss tangent from different curing method of epoxy resins reinforced with 10% 425 µm sawdust

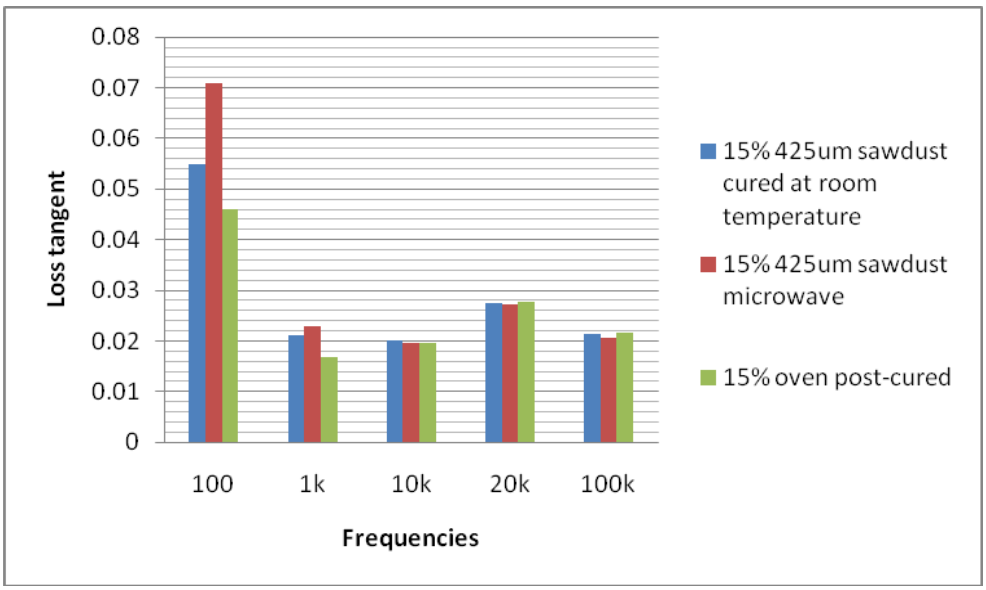


Figure 4.4: Comparison of loss tangent from different curing method of epoxy resins reinforced with 15% 425 µm sawdust

Figure 4.5 shows the loss tangent measured over range of frequencies for the resins mixed with varying percentages of sawdust and cured at room temperature. By viewing Figure 4.14, it can be found that the samples cured at room temperature, pure epoxy resins have the lowest value of loss tangent in the high frequency range (10k Hz and up). The samples which reinforced with 15% and 10% sawdust have the highest value of loss tangent. Therefore, it can be concluded that for the samples cured at room temperature, the loss tangents is increased with the addition of sawdust as fillers.

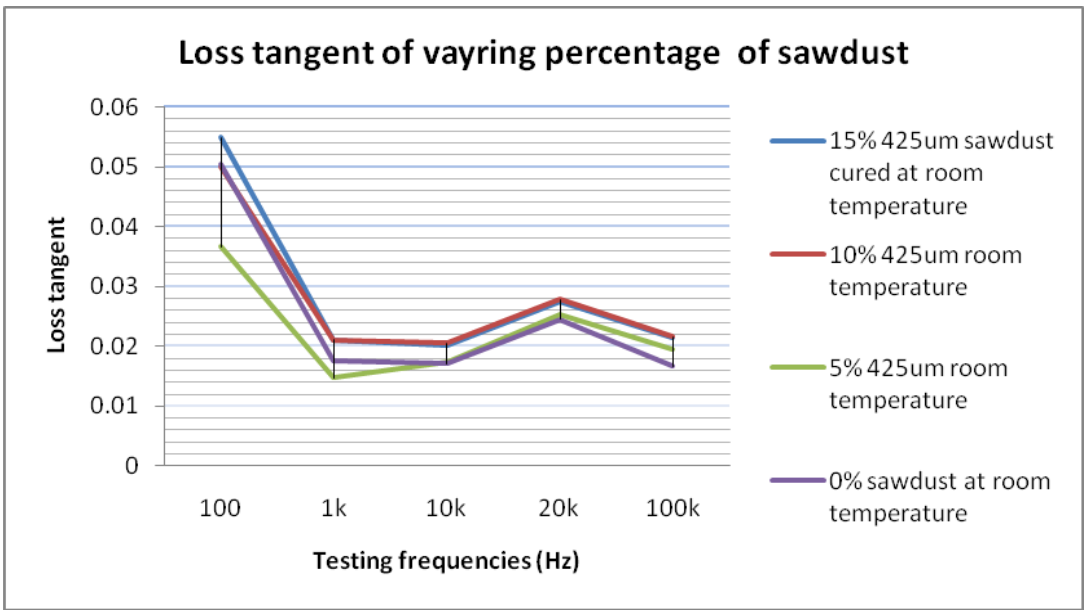


Figure 4.5: Comparison of loss tangent for varying percentage of sawdust cured at room temperature

Figure 4.6 shows the loss tangent measured over range of frequencies for the resins mixed with varying percentages of sawdust and cured with a microwave. The pure epoxy resins are shown in Figure 4.6 have lowest values of loss tangent at all frequencies. These results match with the observation in the laboratory during the microwave heating process. The samples with 15% and 10% sawdust were heated up faster than other samples, hence they needed to be removed from the microwave oven earlier. The results again verified the values of loss tangent were increased by adding sawdust into the epoxy resins.

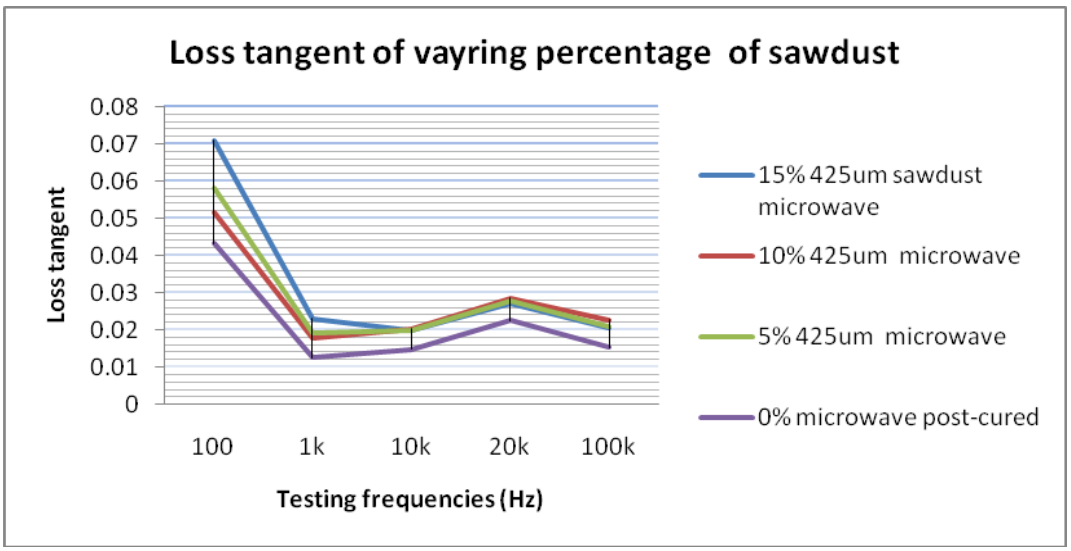


Figure 4.6: Comparison of loss tangent for varying percentage of sawdust cured in microwave

Figure 4.7 shows the loss tangent measured over range of frequencies for the resins mixed with varying percentages of sawdust and post-cured in a conventional oven. Unlike the samples post-cured in the microwave oven, the loss tangent of the oven cured samples displayed no obvious difference between the pure epoxy resins and the sawdust reinforced epoxy resins.

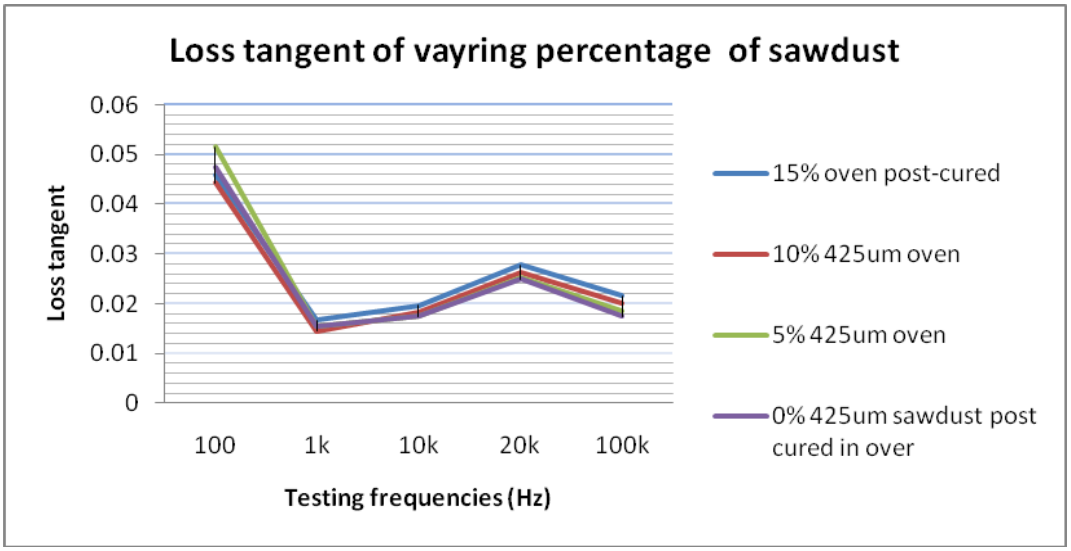


Figure 4.7: Comparison of loss tangent for varying percentage of sawdust cured in oven

4.1.2 The parallel capacitance

Figures 4.8, 4.9 and 4.10 show the measured parallel capacitance of the samples. The capacitance of the samples which are shown in Figures 4.8, 4.9 and 4.10 only decreased slightly as the frequency increase from 100 Hz to 100k Hz. Hence, it can be concluded that the effect of varying frequencies on capacitance of the sample are negligible. As the capacitances of the samples are fairly constant, therefore the samples are able to hold a constant amount of electrical charges over a range of frequencies.

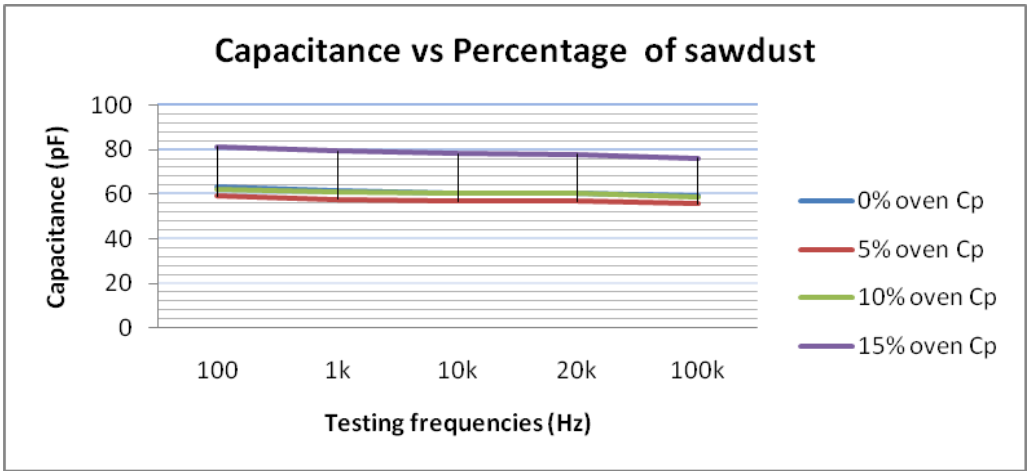


Figure 4.8: Comparison of capacitance for oven cured epoxy resins with varying percentage of sawdust

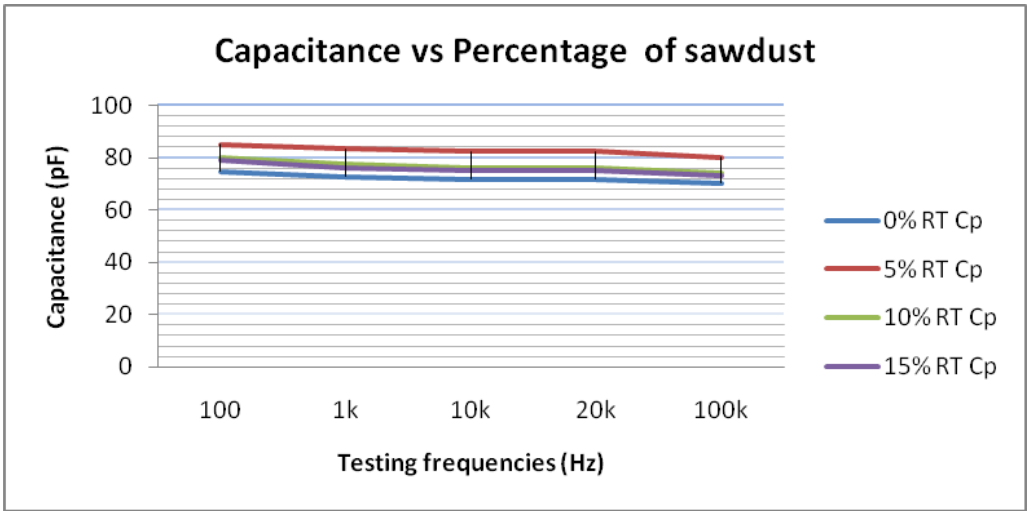


Figure 4.9: Comparison of capacitance for varying percentage of sawdust

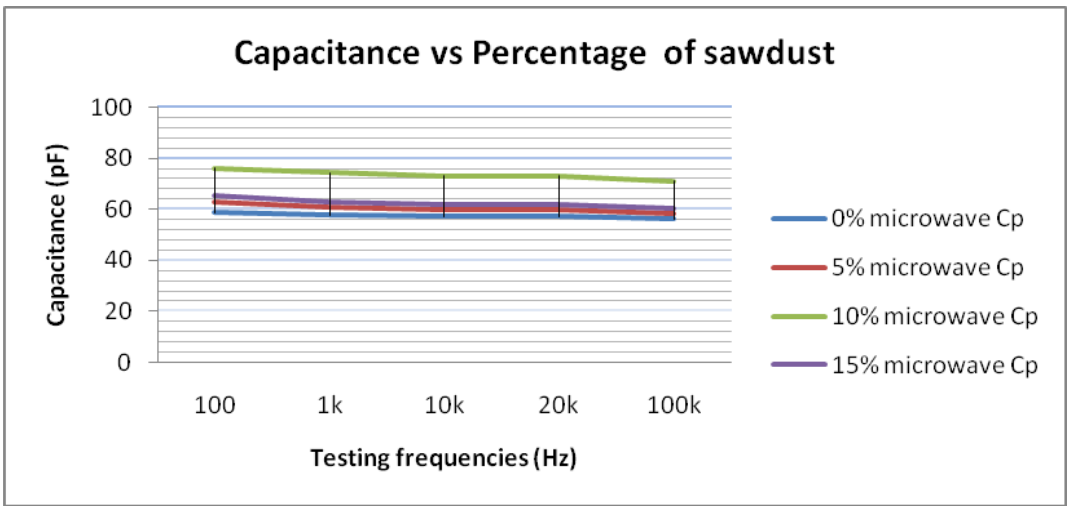


Figure 4.10: Comparison of capacitance for microwave cured epoxy resins with varying percentage of sawdust

4.1.3 Dielectric constant measurement

Figure 4.11 shows the results of measured dielectric constant for sawdust reinforced and oven cured epoxy resin. As shown in Figure 4.11, the general trend for the dielectric constant of the samples was gradually decreased as frequency increased. The sample of neat epoxy resins had the lowest value of dielectric constant over the range of all testing frequencies. The dielectric constant of the sample of neat epoxy resins decreased from 2.763 to 2.595 as the testing frequency increased from 100 Hz to 100 kHz, which decreased 6% in total. The sample of epoxy resin reinforced with 15% sawdust had the highest value of dielectric constant over all range of testing frequencies. By comparing the curves of the dielectric constant of different samples shown in Figure 4.20, it can be concluded that the higher percentage of sawdust added into the resin results in a higher value of dielectric constant of the epoxy composite. The reflection of microwave results at the interface between air and the epoxy composite is proportional to the dielectric constant of the composite. Since the sawdust reinforced epoxy composite had higher value of dielectric constant than pure epoxy composite, it can be deduced that more microwaves will be reflected by the sawdust reinforced composites than the pure epoxy resin. Furthermore, the propagation velocity of microwave is inversely proportional to the square root of the dielectric constant of the medium. Hence, the microwave will move slower in the sawdust reinforced epoxy than in the pure epoxy resins.

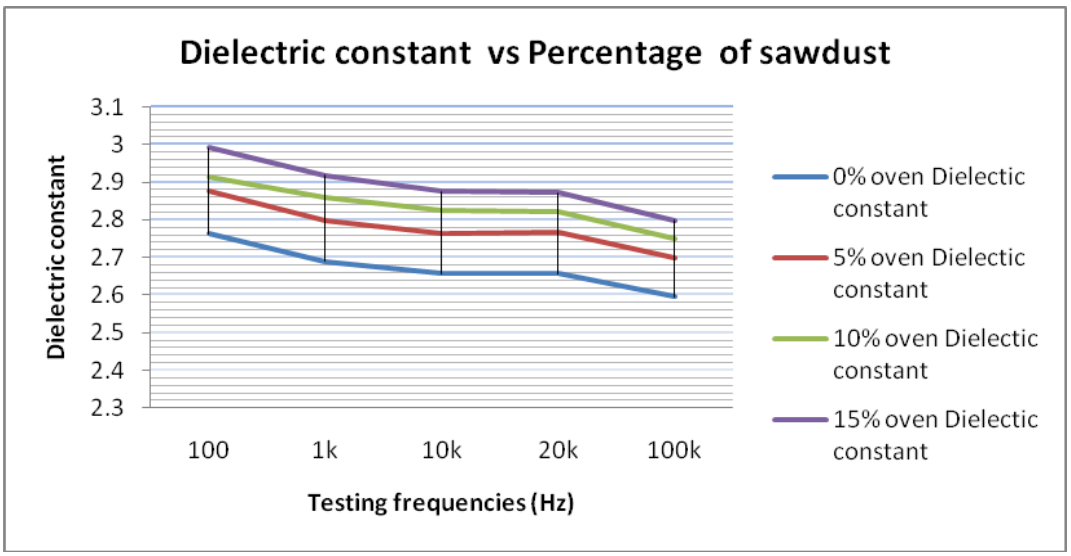


Figure 4.11: Comparison of dielectric constant for oven cured epoxy resins with varying percentages of sawdust

Figure 4.12 shows the dielectric constant for the epoxy resins which were cured at room temperature. By studying the curves in Figure 4.12, it can be found that the pure epoxy resins had the lowest value of dielectric constant over the range of testing frequencies and the epoxy resins reinforced with 15% sawdust had the highest value of dielectric constant over the range of testing frequencies. Hence, the natural cured and sawdust reinforced epoxy resins tend to reflect more microwave than the natural cured neat epoxy resins. Moreover, since the microwave propagation velocity is inversely proportional to the square root of the dielectric constant of the medium, microwaves would move slower in the natural cured and sawdust reinforced epoxy resins than in the natural cured pure epoxy resins.

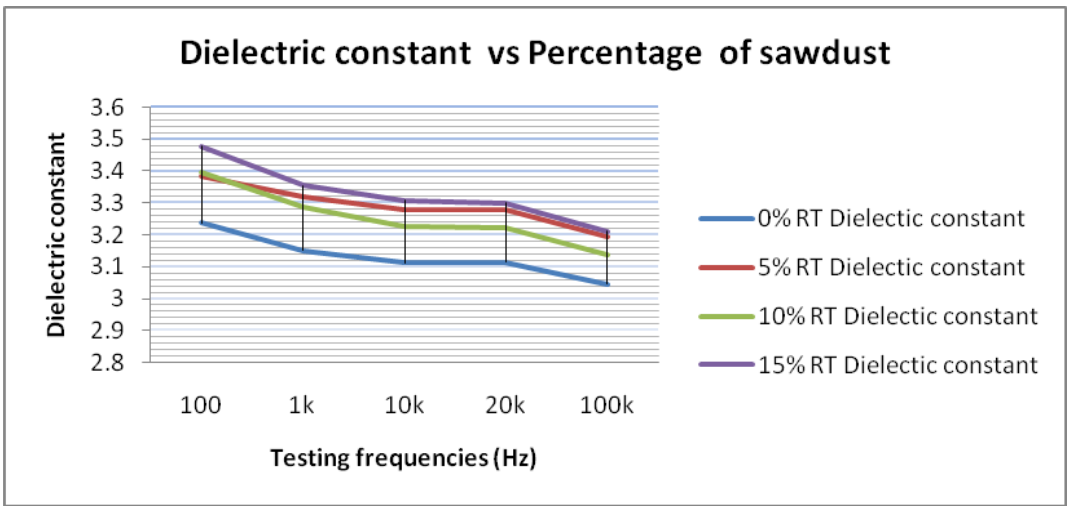


Figure 4.12: Comparison of dielectric constant for epoxy resins cured at room temperature and reinforced with varying percentage of sawdust

Figure 4.13 shows the measured dielectric constant for microwave cured epoxy resins. The microwave cured neat epoxy resins again had the lowest value of dielectric constant and the epoxy resins reinforced with 15% sawdust have the highest value of dielectric constant. Thus, it can be concluded that the microwave cured and sawdust reinforced epoxy resins tend to reflect more microwave than the microwave cured neat epoxy resins. Considering Figure 4.11, 4.12 and 4.13, it can be found that the dielectric constant increased with increasing percentage of sawdust added into the epoxy resins.

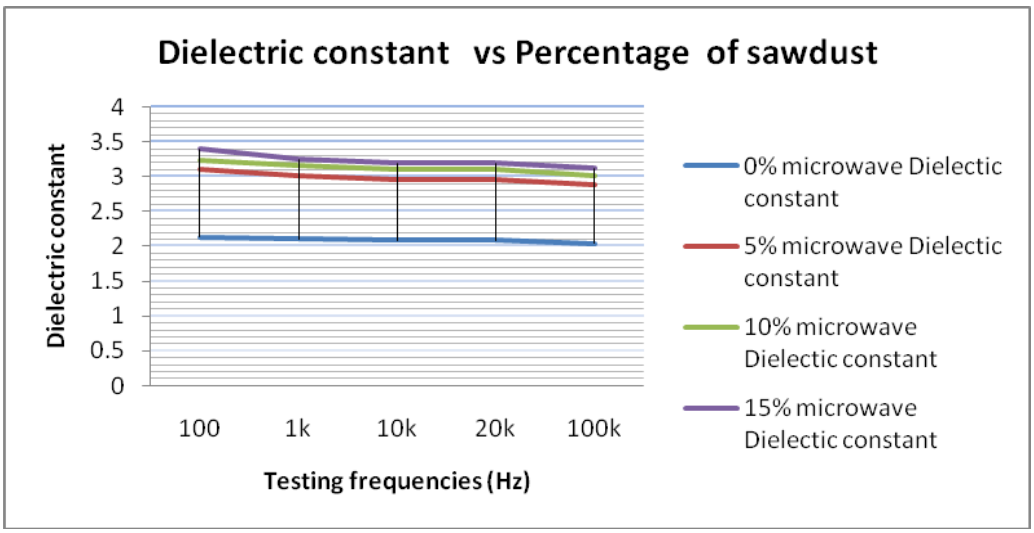


Figure 4.13: Comparison of dielectric constant for microwave cured epoxy resins with varying percentage of sawdust

Figure 4.14 shows the dielectric constant for the neat epoxy resins which were treated with different curing methods. By comparing the results shown in Figure 4.14, it can be found that the microwave cured pure epoxy resins had the lowest dielectric constants over the range of the testing frequencies and the natural cured epoxy resins had the highest value of dielectric constant.

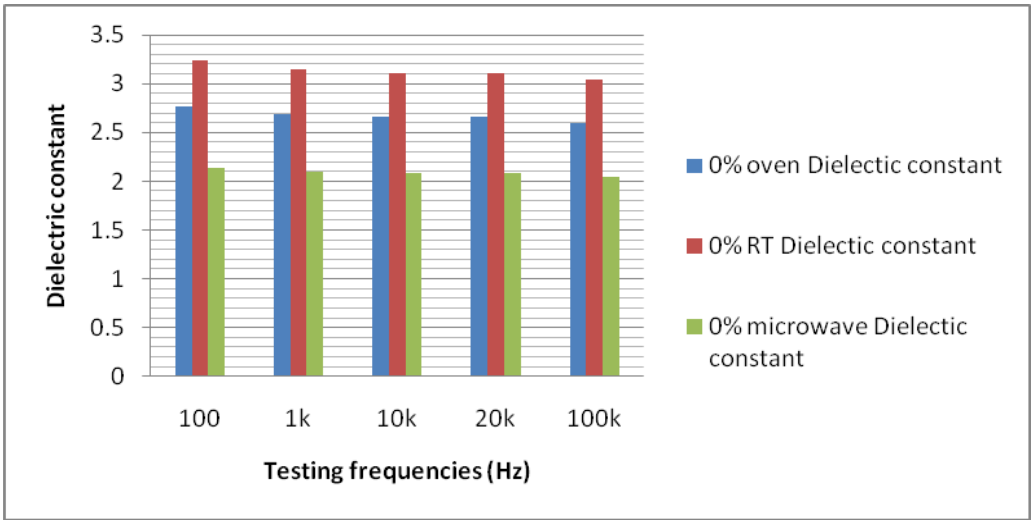


Figure 4.14: Comparisons of the dielectric constant for neat epoxy resins

With reference to Figures 4.15, 4.16 and 4.17, it can be found that the oven cured epoxy resins had the lowest dielectric constants over the range of the testing frequencies and the natural cured epoxy resins had the highest value of dielectric constant over the range of the testing frequencies. Hence, the microwave cured epoxy resins will reflect more microwave than oven cured epoxy resins and natural cured epoxy resins will reflect more microwave than microwave cured epoxy resins.

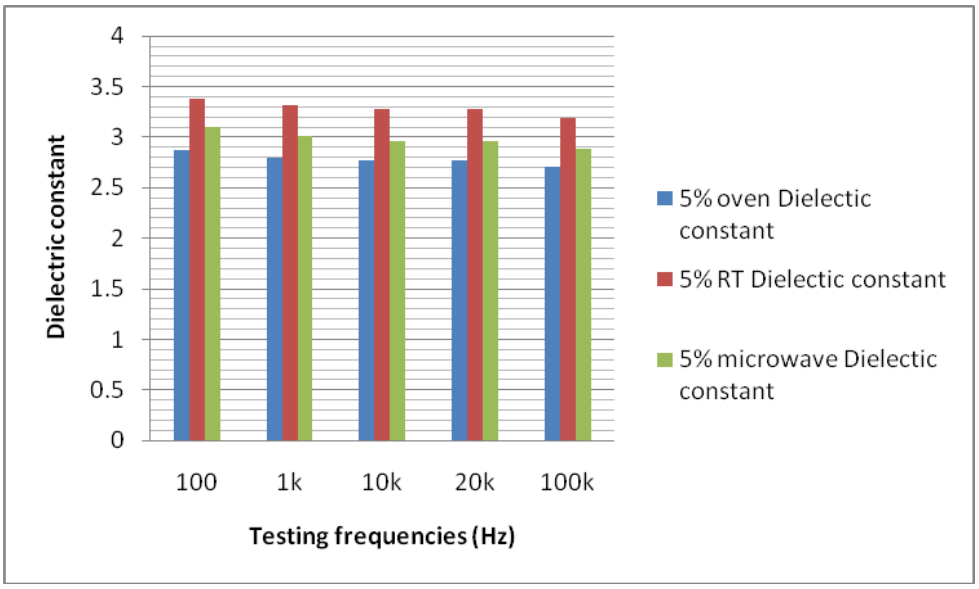


Figure 4.15: Comparisons of the dielectric constant for epoxy resins reinforced with 5% sawdust

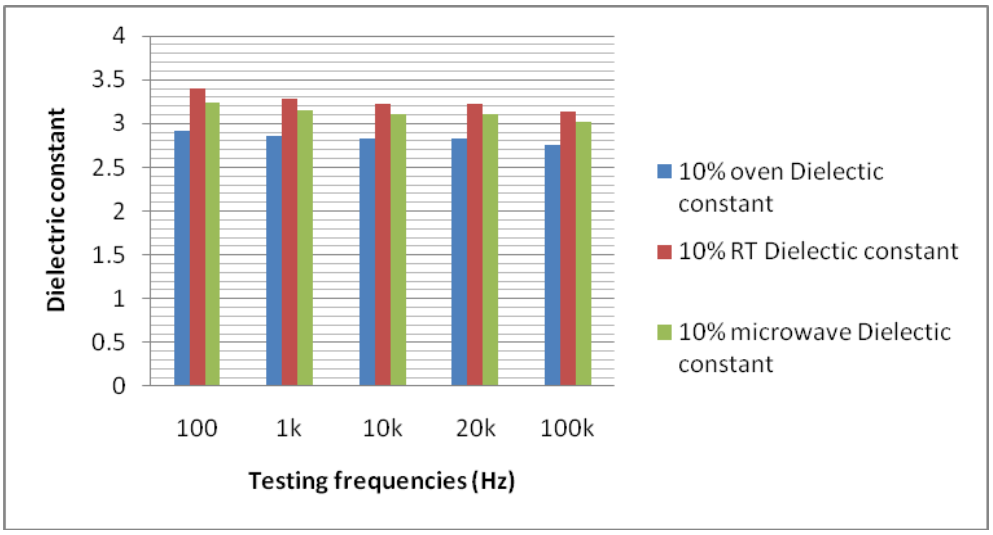


Figure 4.16: Comparisons of the dielectric constant for epoxy resins reinforced with 10% sawdust

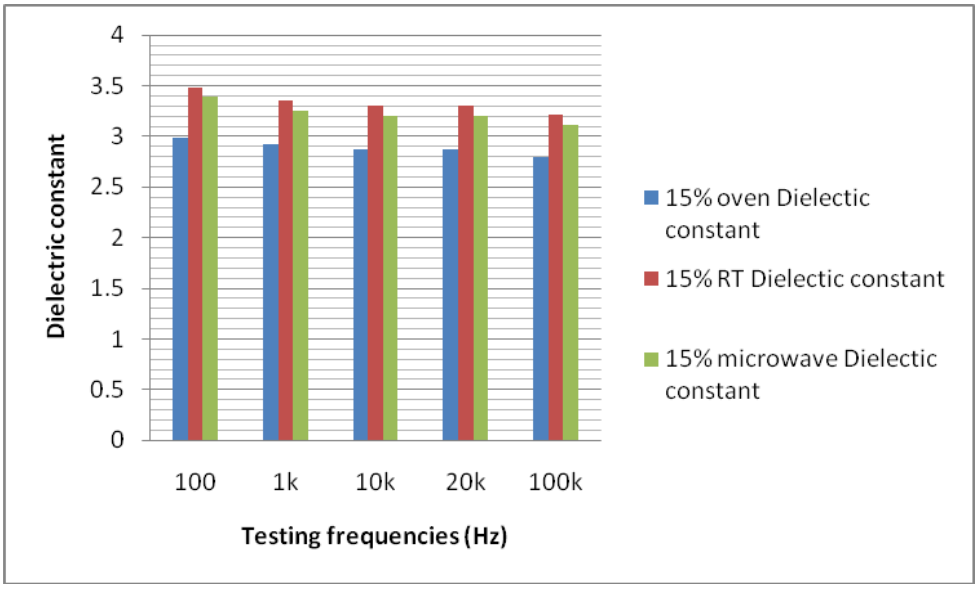


Figure 4.17: Comparisons of the dielectric constant for epoxy resins reinforced with 15% sawdust

4.2 DMA test results

Figures 4.18, 4.19 and 4.20 show the glass transition temperatures for naturally cured, microwave cured and oven cured samples were 65.92 ^{o}C, 75.69 ^{o}C and 83.82 ^{o}C respectively. These figures also illustrate that the storage moduli of them are 1341MPa, 1457 MPa and 1815 MPa respectively. The loss factor tanδ for naturally cured, microwave cured and oven cured were 1.4, 1.05 and 1.04 respectively. Since the loss factor tan δ is the ratio of loss modulus E'' to storage modulus E', loss modulus E'' can be easily calculated once E' and tanδ are known. The calculated loss modulus E'' for naturally cured, microwave cured and oven cured were 1877.4 MPa, 1529.85 MPa and 1887.6 MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus and loss modulus indicate the material is softer and has a higher water content and less degree of cure. Since the oven cured sample has the highest glass transition temperature and storage modulus, it is the stiffest sample. The oven cured sample also has the highest loss modulus, theoretically, for the sample has high storage modulus should have low loss modulus, but this is not the case. However, the oven cured sample has the lowest loss factor tanδ which means the oven

cured sample is stiffest and have highest degree of cure. That is consistent with the conclusion drawn from the glass transient temperature.

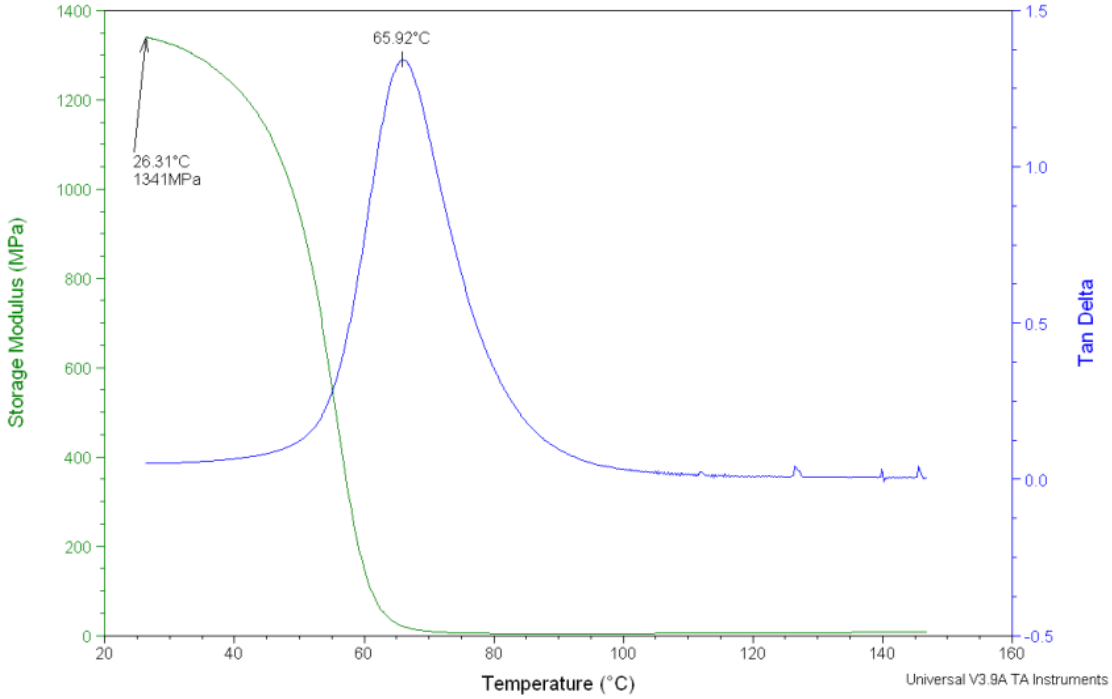


Figure 4.18: DMA test results for naturally cured epoxy resins reinforced with 5% 425 µm sawdust

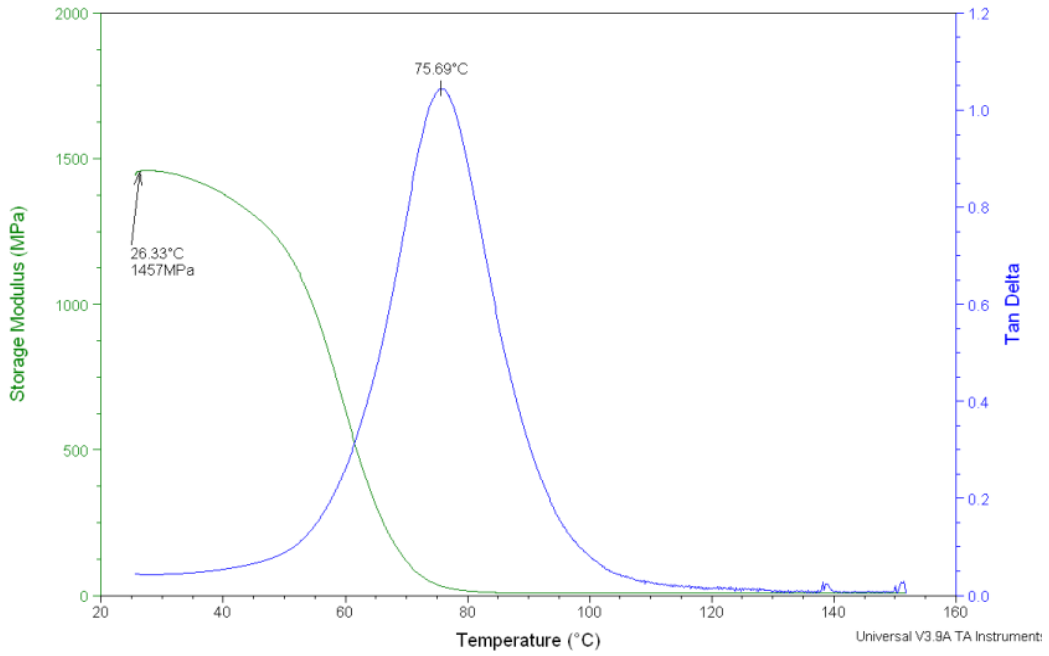


Figure 4.19: DMA test results for microwave cured epoxy resins reinforced with 5% 425 µm sawdust

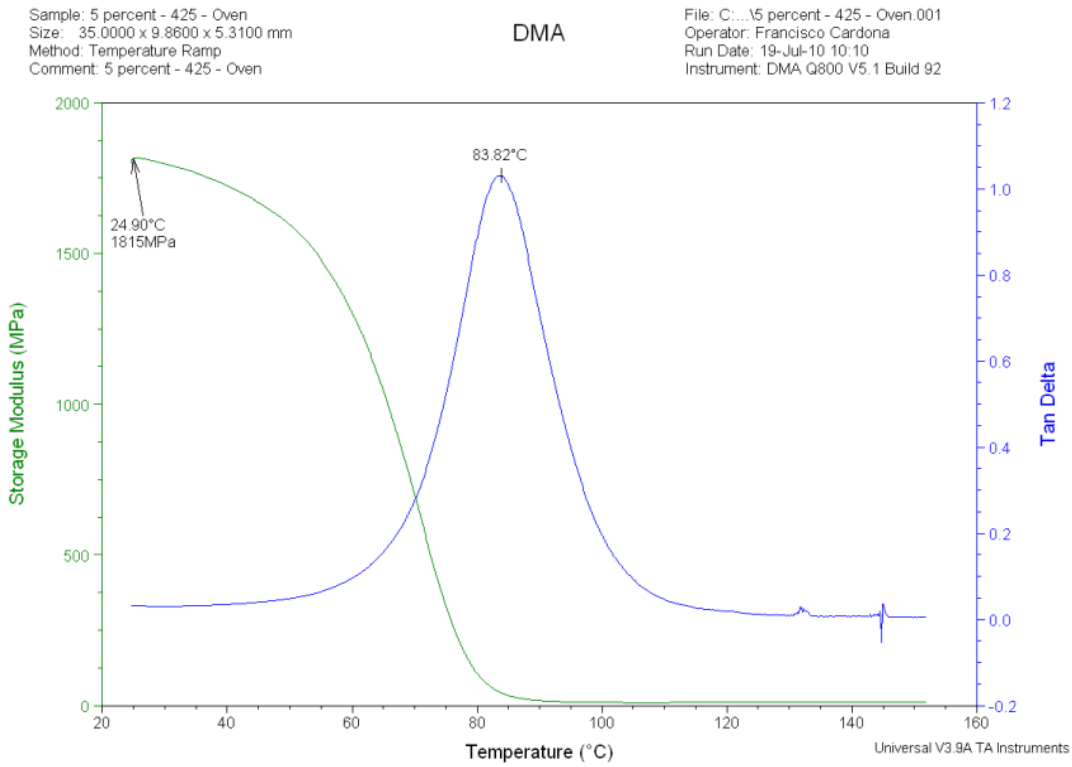


Figure 4.20: DMA test results for oven cured epoxy resins reinforced with 5% 425 µm sawdust

Figure 4.21, 4.22 and 4.23 show the glass transition temperatures for naturally cured, microwave cured and oven cured samples were 62.81 ^{o}C, 74.97 ^{o}C and 83.27 ^{o}C respectively. These figures also illustrate that the storage moduli of them are 1283 MPa, 1392 MPa and 1967 MPa respectively. The loss factor tanδ for naturally cured, microwave cured and oven cured were 1.38, 0.98 and 0.96 respectively. Since the loss factor tan δ is the ratio of loss modulus E'' to storage modulus E', loss modulus E'' can be easily calculated once E' and tanδ are known. The calculated loss modulus E'' for naturally cured, microwave cured and oven cured were 1770.54 MPa, 1364.16 MPa and 1888.32 MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus and loss modulus indicate the material is softer and has a higher water content and less degree of cure. Since the oven cured sample has the highest glass transition temperature and storage modulus, it is the stiffest sample. The oven cured sample also has the highest loss modulus, theoretically, for the sample has high storage modulus should have low loss modulus, but this is not the case. However, the oven cured sample has the lowest loss factor tanδ which means the oven cured sample is stiffer. This result is consistent with the conclusion draw from the glass transient temperature.

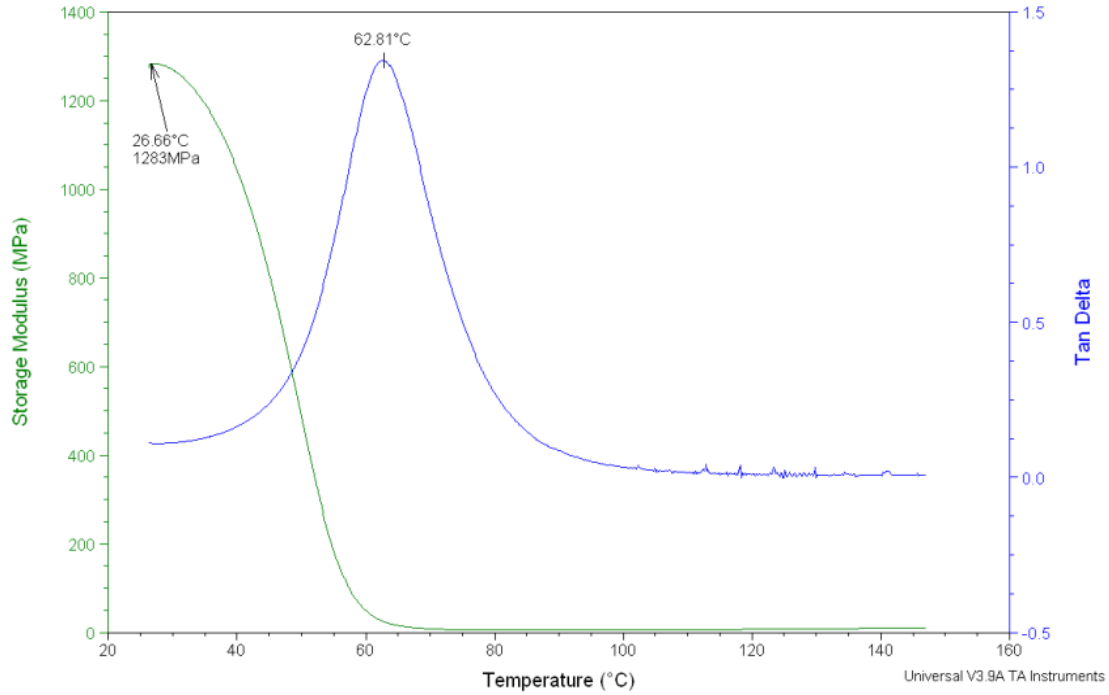


Figure 4.21: DMA test results for naturally cured epoxy resins reinforced with 10% 425 µm sawdust

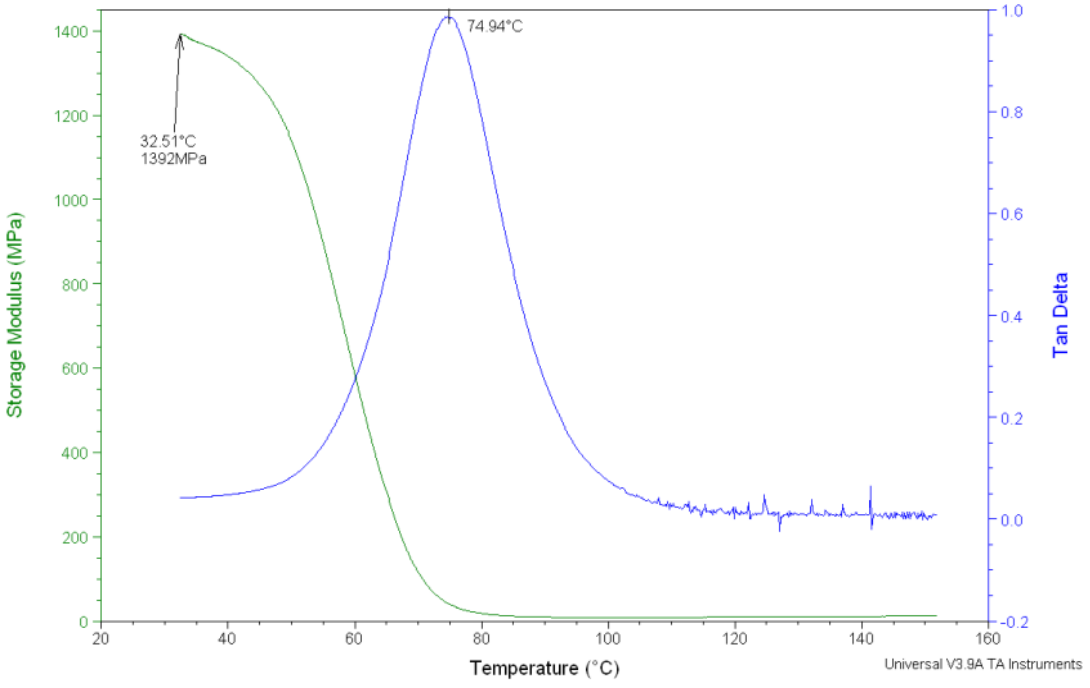


Figure 4.22: DMA test results for microwave cured epoxy resins reinforced with 10% 425 µm sawdust

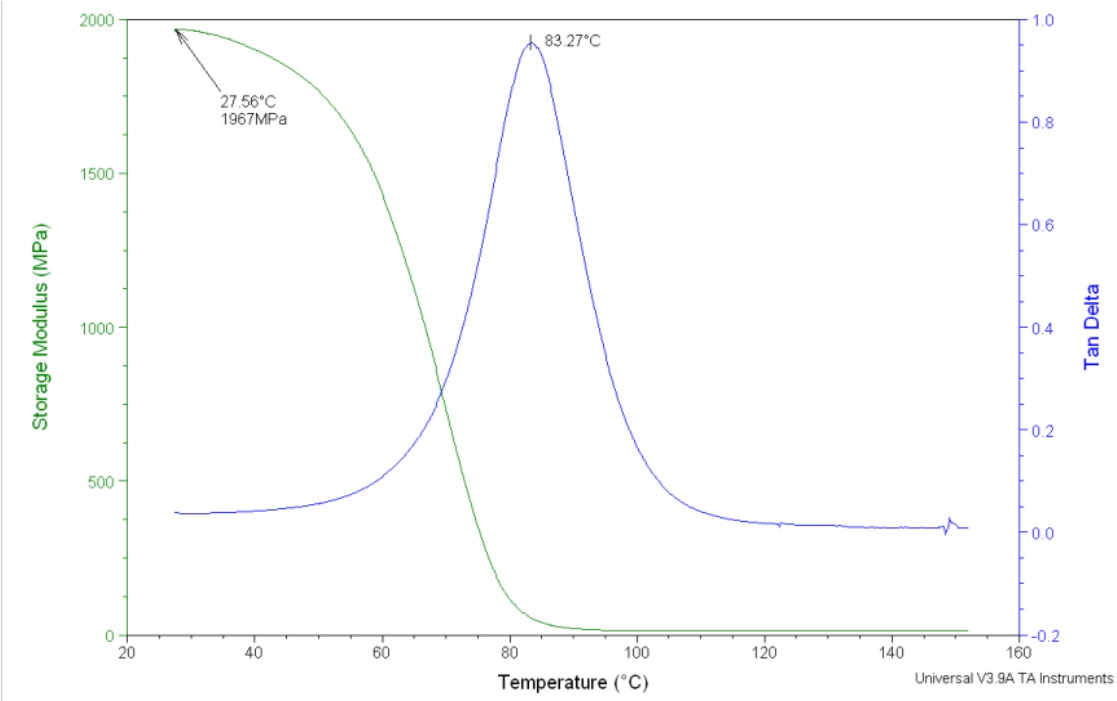


Figure 4.23: DMA test results for oven cured epoxy resins reinforced with 10% 425 µm sawdust

Figure 4.24, 4.25 and 4.26 show the glass transition temperatures for naturally cured, microwave cured and oven cured samples were 61.41 ^{o}C, 68.39 ^{o}C and 83.6 ^{o}C respectively. These figures also illustrate that the storage moduli of them are 2656 MPa, 1728 MPa and 1964 MPa respectively. The loss factor tanδ for naturally cured, microwave cured and oven cured were 1.15, 1.05 and 0.86 respectively. Since the loss factor tan δ is the ratio of loss modulus E'' to storage modulus E', loss modulus E'' can be easily calculated once E' and tanδ are known. The calculated loss modulus E'' for naturally cured, microwave cured and oven cured were 3054.4 MPa, 1814.4 MPa and 1689.04 MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus and loss modulus indicate the material is softer and have higher water content and less degree of cure. Since the oven cured sample has the highest glass transition temperature, it can withstand the highest operating temperature and still be able to retain reasonable mechanical strength. The naturally cured sample has the highest storage modulus which means it is the stiffest sample. However, the naturally cured sample also has the highest loss modulus and lowest loss factor tanδ, theoretically, for the sample has high storage modulus should have low loss modulus and lowest loss factor tanδ, but this is not the case.

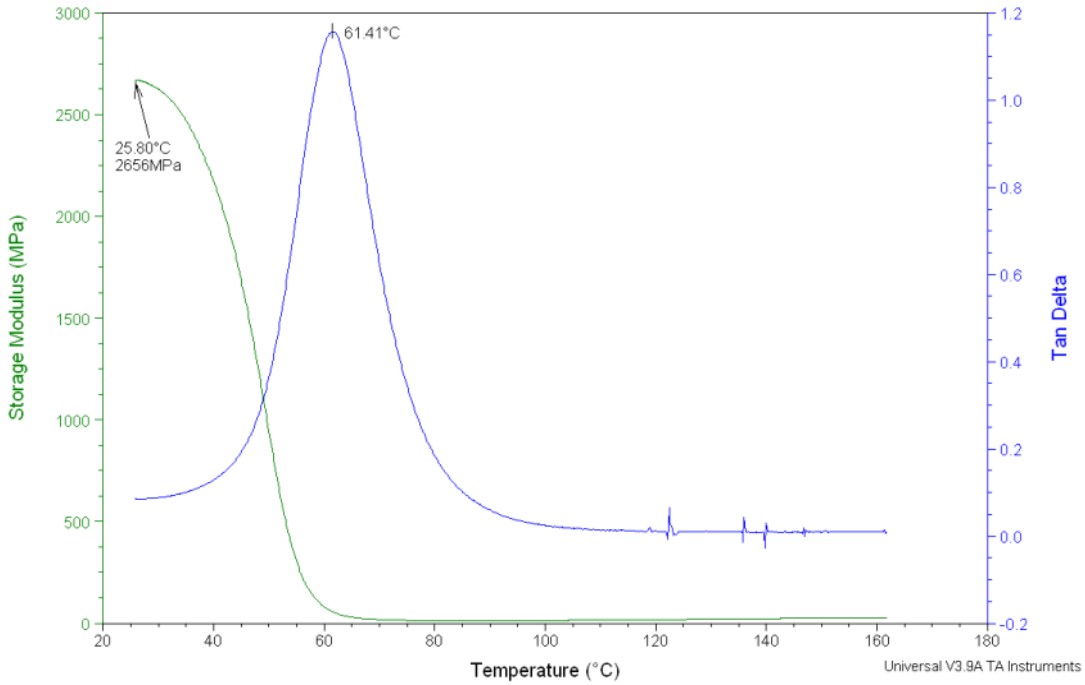


Figure 4.24: DMA test results for naturally cured epoxy resins reinforced with 15% 425 µm sawdust

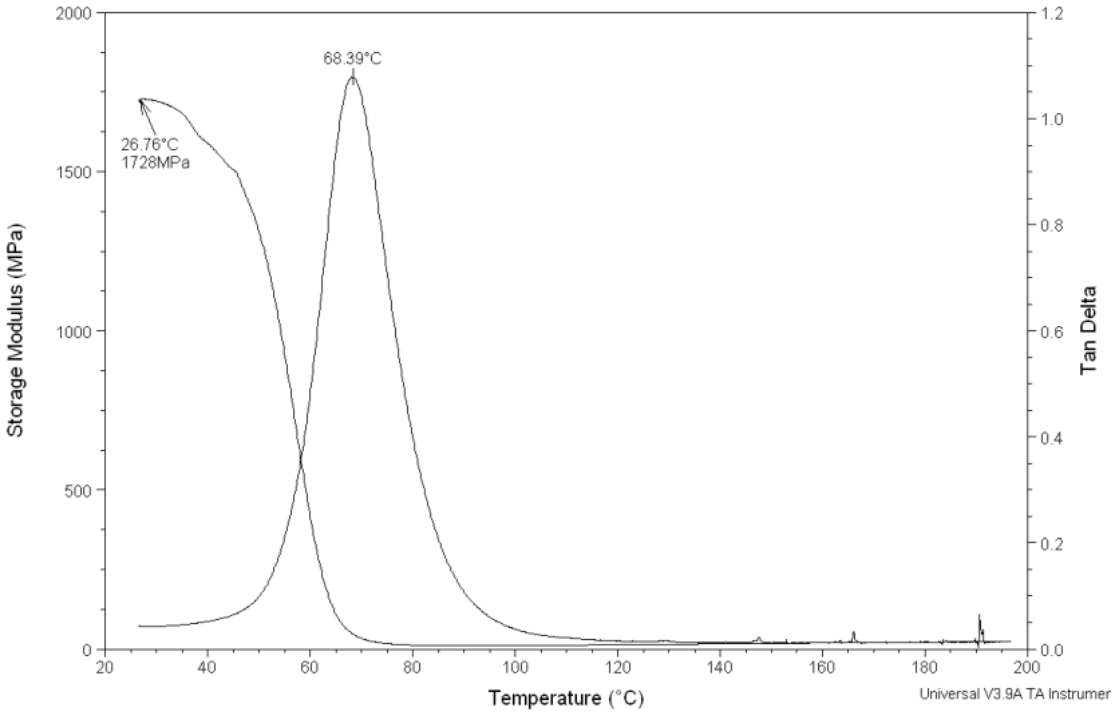


Figure 4.25: DMA test results for microwave cured epoxy resins reinforced with 15% 425 µm sawdust

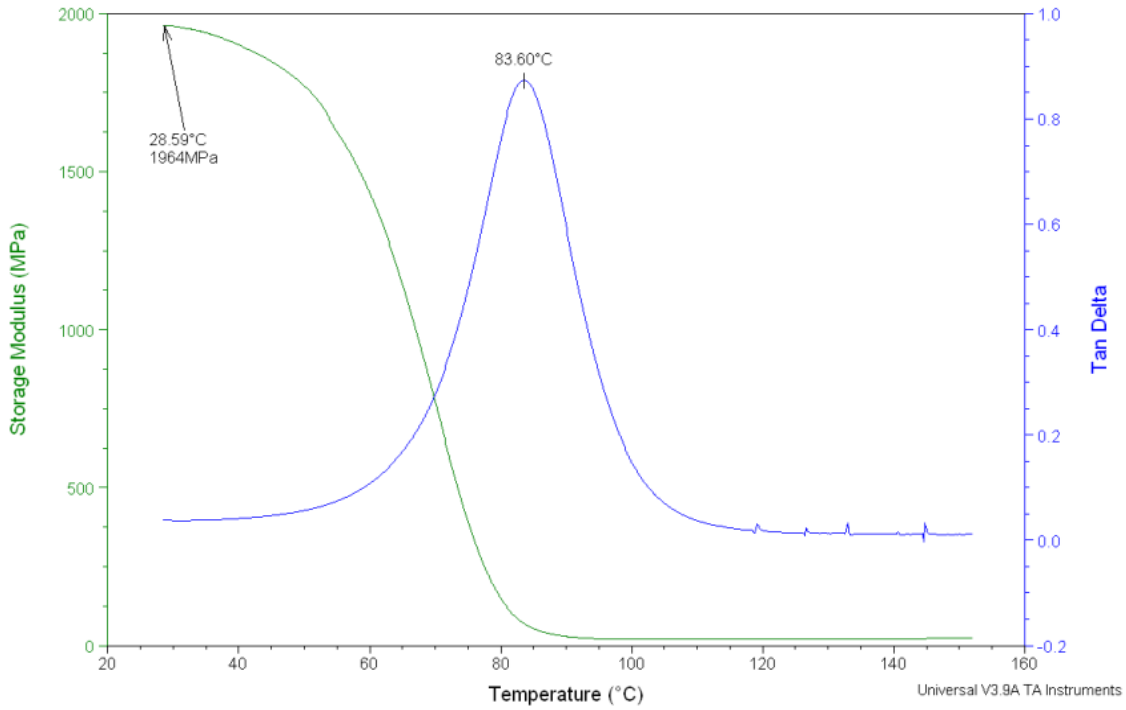


Figure 4.26 DMA test results for oven cured epoxy resins reinforced with 15% 425 µm sawdust

Figure 4.27, 4.28 and 4.29 show the glass transition temperatures for naturally cured, microwave cured and oven cured samples were 66.69 ^{o}C, 70.93 ^{o}C and 80.87 ^{o}C respectively. These figures also illustrate that the storage modulus of them are 1427 MPa, 1070 MPa and 1580 MPa respectively. The loss factor tanδ for naturally cured, microwave cured and oven cured were 1.4, 1.05 and 1.15 respectively. Since the loss factor tan δ is the ratio of loss modulus E'' to storage modulus E', loss modulus E'' can be easily calculated once E' and tanδ are known. The calculated loss modulus E'' for naturally cured, microwave cured and oven cured were 1997.8 MPa, 1123.5 MPa and 1817 MPa respectively. Higher glass transition temperature and high storage modulus mean stiffer material. Higher loss modulus and loss modulus indicate the material is softer and have higher water content and less degree of cure. Since the oven cured sample has the highest glass transition temperature and storage modulus, it is the stiffest sample. The naturally cured sample has the highest loss modulus and lowest glass transition temperature, therefore it is the softest sample. Theoretically, for the sample has highest loss modulus should have lowest storage modulus, but this is not the case.

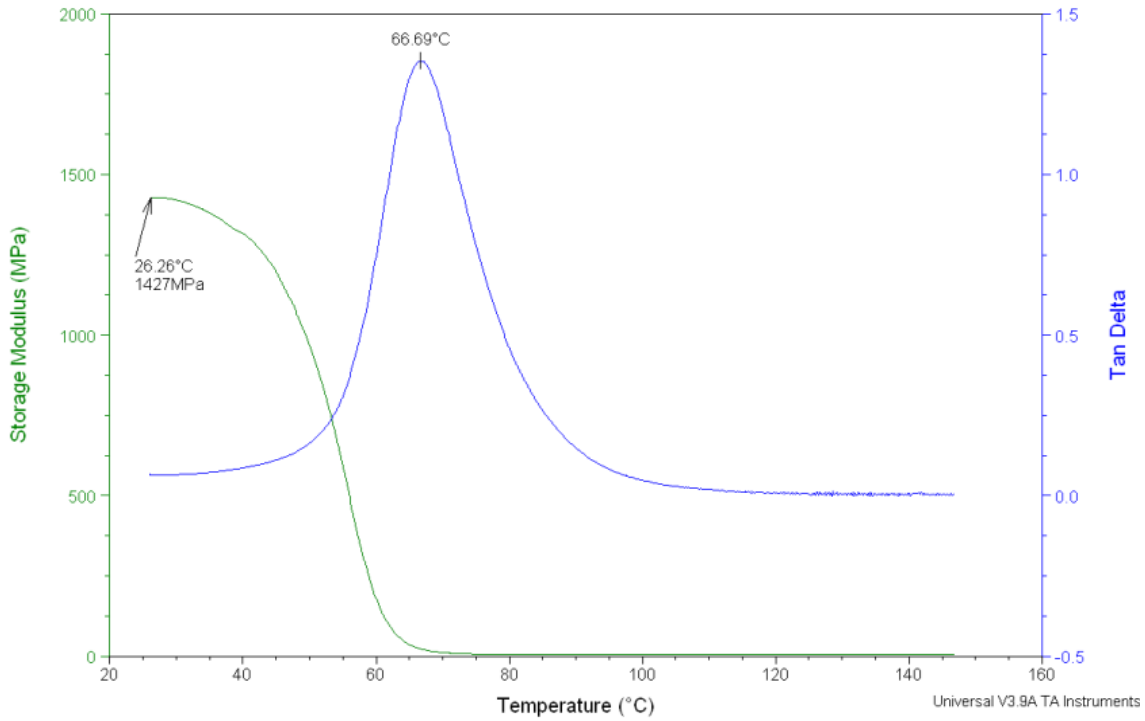


Figure 4.27: DMA test results for naturally cured epoxy resins

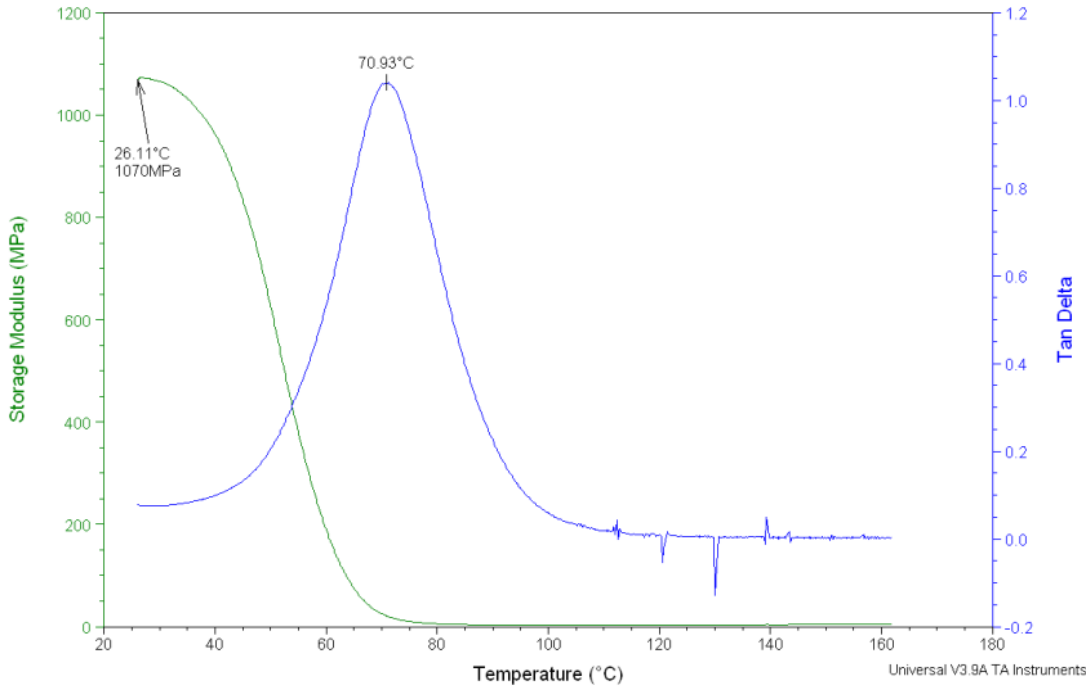


Figure 4.28: DMA test results for microwave cured neat epoxy resins

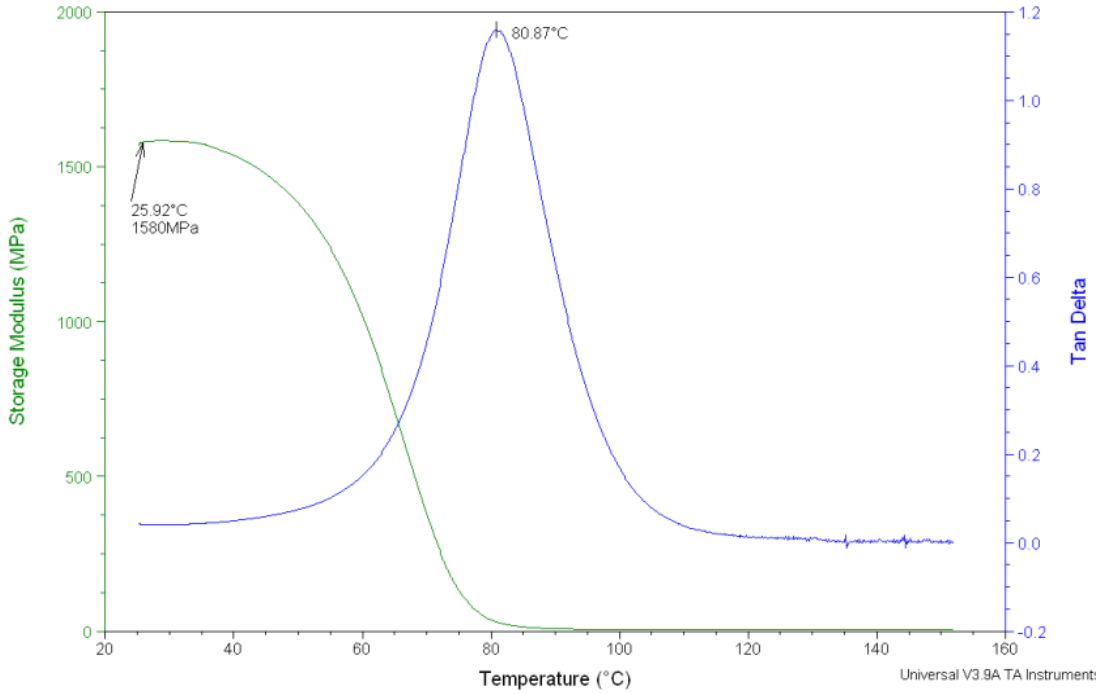


Figure 4.29: DMA test results for oven cured pure epoxy resins

4.2.1 DMA test summary

Table 4.1 shows the summary of DMA test results. It included the results for maximum loss modulus E'', maximum storage modulus E' and maximum loss factor tanδ. It can be found from Table 4.1 that the oven cured samples generally have the highest glass transient temperature in all of the test samples.

Table 4.1: DMA test results

Samples cured in oven				
Percentage of sawdust	Glass transient temperature Tg (^{o}C)	Maximum Storage modulus E' (MPa)	Maximum loss factor tanδ	Maximum loss modulus
0%	80.87	1580	1.15	1817
5%	83.82	1815	1.04	1887.6
10%	83.27	1967	0.96	1888.32
15%	83.6	1964	0.86	1689.04
Samples cured in microwave				
Percentage of sawdust	Glass transient temperature Tg (^{o}C)	Maximum Storage mudulusE'(MPa)	Maximum loss factor tanδ	Maximum loss mudulus
0%	70.93	1070	1.05	1123.5
5%	75.69	1457	1.05	1529.85
10%	74.97	1392	0.98	1364.16
15%	68.39	1728	1.05	1814.4
Samples cured at room temperature				
Percentage of sawdust	Glass transient temperature Tg (^{o}C)	Maximum Storage mudulusE'(MPa)	Maximum loss factor tanδ	Maximum loss mudulus
0%	66.69	1427	1.4	1997.8
5%	65.92	1341	1.4	1877.4
10%	62.81	1283	1.38	1770.54
15%	61.41	2656	1.15	3054.4

Figure 4.30 shows the glass transition temperature for oven cured, microwave cured and naturally cured epoxy resins reinforced with 5%, 10% and 15% of sawdust. By comparing the results, it can be found that the oven cured samples in general have the highest glass transient temperature in the group of test samples. The microwave cured samples have the moderate glass transient temperature in the group of test samples. The naturally cured samples have the lowest glass transient temperature in the group of test samples. The results mean the oven cured samples can withstand higher temperature and still are capable of retaining reasonable mechanical strength than naturally cured

and microwave cured samples. Since all three curves in Figure 4.30 are flat, it can be concluded that the additions of sawdust do not have significant effect on the glass transition temperature of epoxy resins.

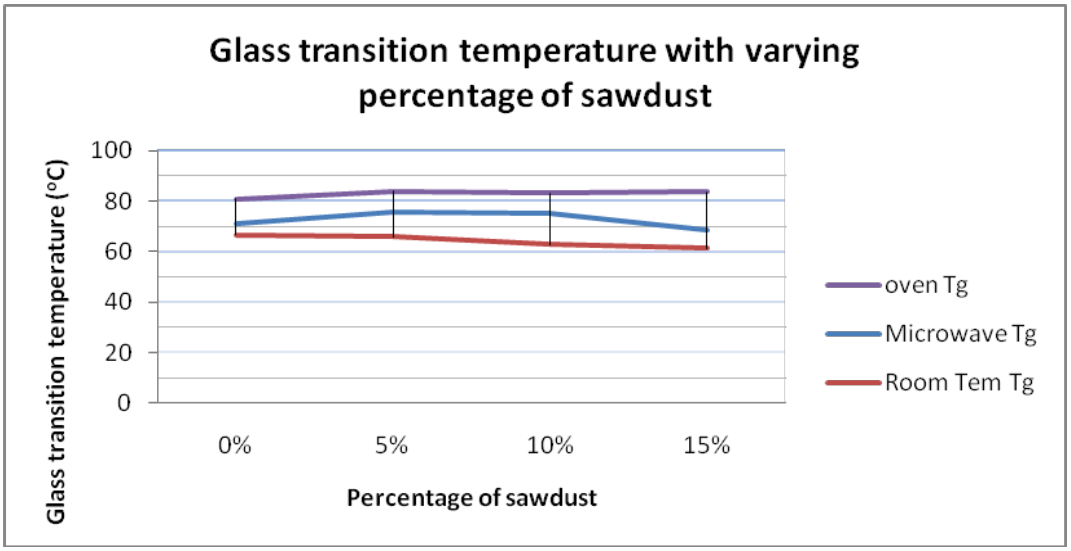


Figure 4.30: Glass transition temperature of epoxy resins

Figure 4.31 shows the storage modulus for oven cured, microwave cured and naturally cured epoxy resins reinforced with 5%, 10% and 15% of sawdust. As shown in figure 4.31, oven cured samples have higher storage modulus than microwave cured samples. However, it is difficult to compare the storage modulus of naturally cured samples with other samples', since the storage modulus of naturally cured samples does not have a consistent trend.

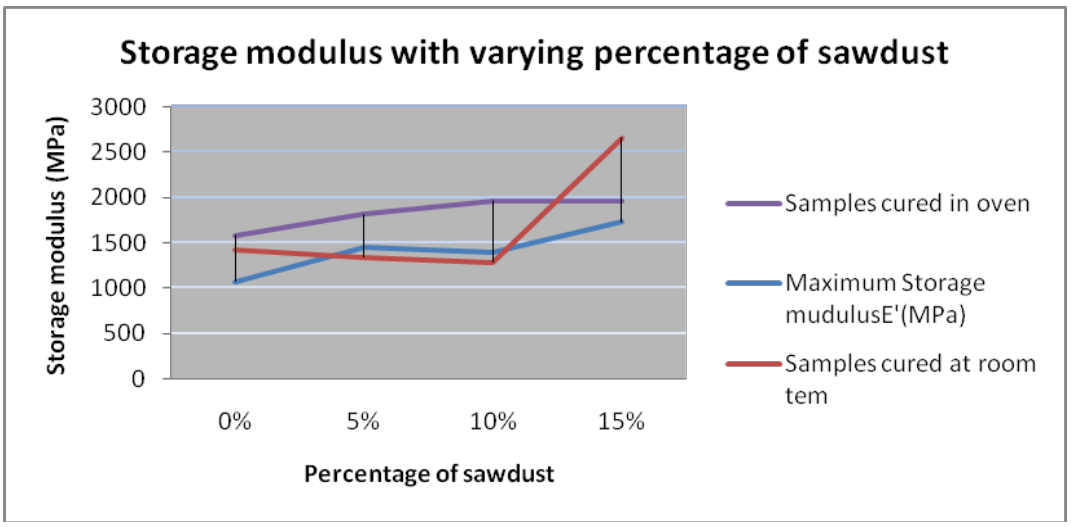


Figure 4.31: Storage modulus of epoxy resins

Figure 4.32 shows the loss modulus for oven cured, microwave cured and naturally cured epoxy resins reinforced with 5%, 10% and 15% of sawdust. As shown in figure 4.32, oven cured samples and the naturally cured samples have higher loss modulus than microwave cured samples. That means oven cured samples and naturally cured samples have less degree of cure than microwave cured samples.

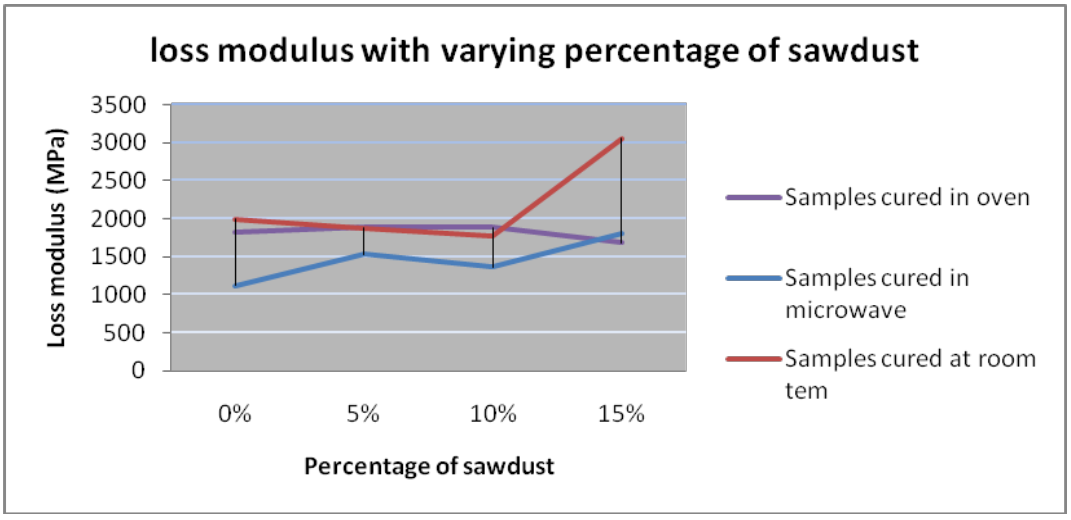


Figure 4.32: Loss modulus of epoxy resins

4.3 Tensile test results

4.3.1 Example calculation

Example for calculating yield strength, tensile strength and Young's modulus are given below (the specimen 3 of 10% 425µm sawdust is used here for the calculation demonstration):

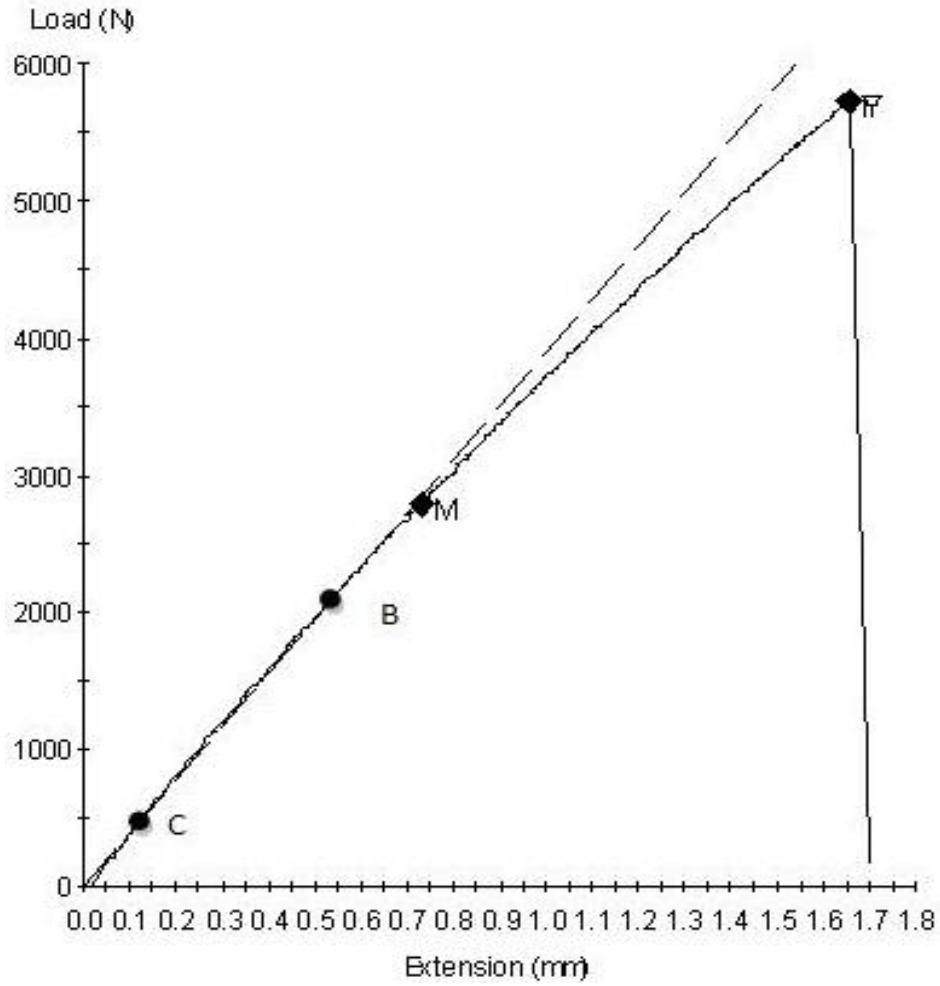


Figure 4.33: the load and extension curve for the specimen 3 of 10% 425µm sawdust sample set

Figure 4.33 shows the load and extension curve used for this example calculation. From Figure 4.33, the following data can be found:

The thickness of the test specimen: 8.05 mm

The width of the test specimen: 25.00mm

Gauge length L: 150 mm

Cross- sectional area A = 8.05 × 25.00 = 201.25 mm^2

In order to obtain the accurate value of the Young's Modulus E, the slope of the most linear part of the load – extension curve should be calculated. Hence, point C (load is at 485.6 N and extension is at 0.127 mm) and point B (load is at 2003 N and extension is at 0.513 mm) are selected for the slope calculation. Thus, F = 2003– 485.6 = 1517.4 N and ΔL = 0.513 – 0.127 = 0.386 mm. Substituting these values into Eq. (2.3), it would work out the corresponding value of the Young's modulus:

Stress = $\frac{F}{A} = \frac{1517.4}{201.25}$ = 7.540 MPa

Strain = $\frac{\Delta L}{L} = \frac{0.386}{150}$ = 2.573 × 10^{-3}

E = $\frac{stress}{strain} = \frac{7.540}{2.573 \times 10^{-3}}$ = 2930.05 MPa

The tensile strength can be calculated by substituting the measured peak load which is 5730 N in this case into Eq. (2.2):

Tensile strength = $\frac{Maximum\ load\ (F)}{Original\ cross-sectional\ area\ (A)} = \frac{5730}{201.25} = 28.47$ MPa

Similarly, the Yield strength also can be calculated by substituting the measured yield load which is 2838.88 N into Eq. (2.1):

Yield strength = $\frac{Yield\ load\ (F)}{Original\ cross-sectional\ area\ (A)} = \frac{2838.88}{201.25} = 14.106$ MPa

4.3.2 Test results for the composites filled with 300 µm sawdust

Table 4.2shows the row data of the test results for the specimens which filled up with 5% 300 µm sawdust. There are four pieces of test specimens are made for each size of sawdust composites. The peak stress is the tensile strength and the modulus is the Young's modulus. Some of the results are far from the mean value and seems to be in error. This data was discarded from the mean value calculation. For example, the peak stress of specimen 1 is 6.69 MPa (highlight in red), it is far away from the peak stress of other specimens in the table, and hence it has been ignored from the mean calculation. There are several possible reasons to cause specimen 1 to have such a low value of tensile stress. One possibility is there are excessive amounts of air bubbles existing in specimen 1, this possibility can be verified by the microscopy inspection. Another reason could be the over-heating caused by the hot spot in the microwave oven, thus damaging the specimen. All the row data have been processed in a similar manner.

Table 4.2: Row data record by computer

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.150	26.880	192	1286	6.69	1286	6.69
2	7.040	26.040	183	4964	27.08	4964	27.08
3	7.100	24.990	177	6805	38.35	6805	38.35
4	7.130	24.990	178	4980	27.95	4980	27.95
Mean	**7.105**	**25.725**	**183**	**4509**	**25.02**	**4509**	**25.02**
Std Dev	**0.048**	**0.915**	**7**	**2316**	**13.25**	**2316**	**13.25**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.342	5.726	1100.436	3076			
2	1.767	13.727	2516.434	2554			
3	2.654	17.868	3170.385	2665			
4	1.939	13.343	2377.453	2431			
Mean	**1.676**	**12.666**	**2291.177**	**2681**			
Std Dev	**0.969**	**5.060**	**865.843**	**280**			

Table 4.3 shows the processed mean values of the test results for varies percentage of sawdust with diameters of 300 μm.

Table 4.3: Processed mean values of the test results for samples reinforced with 300 μm sawdust

Samples filled with 300 μm sawdust					
Percentage of saw dust	0%	5%	10%	15%	16.70%
Young's modulus (MPa)	2521	2550	2635	3004	2994
Tensile strength (MPa)	46.81	30.83	31.93	28.21	29.94
0.05% offset Yield strength (MPa)	21.615	14.97933	15.008	14.219	15.58467

Figures 4.34, 4.35 and 4.36 are produced base on the processed data listed in Table 4.3. As listed in Table 4.3, the Young's modulus for the sample without sawdust (0% sawdust) was at 2521 MPa which is the lowest value in the group. The Young's modulus then continually increased until the percentage of sawdust reached at 15%. The highest value of Young's modulus was found to be 3004 MPa at 15% of sawdust which was 20% higher than the value of pure resin. The maximum percentage of sawdust can mix with epoxy resins are 16.7%, any more sawdust added into the composites would be too stick to mix with. The value of Young's modulus then dropped slightly to 2994 MPa at 16.7% of sawdust. The trend generally shows that the higher the percentage of sawdust in the composites the higher value of the Young's modulus. However, this trend changed when the percentage of sawdust reached 16.7%. The reason for that may be because the excessive amount of sawdust presented in the composites started weakening the adhesion strength between the epoxy resins and the sawdust.

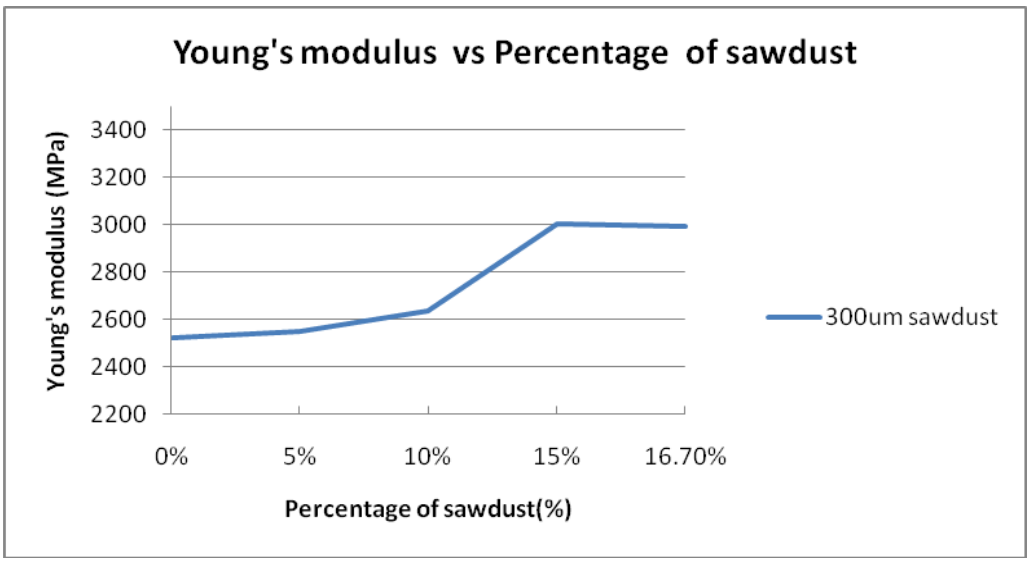


Figure 4.34: Young's modulus versus percentage of 300 µm sawdust

Figure 4.35 depicts the tensile strength of the samples filled with 300 µm sawdust. It was found that the highest tensile strength was 46.81 MPa at 0% of sawdust. The value of the tensile strength then fell dramatically to 30.83 MPa at 5% of sawdust. After this point, the value of tensile strength fluctuated slightly between 31.93 and 28.21 MPa. Hence, it can be concluded that the present of sawdust as fillers improved the Young's modulus of the epoxy composites, but the sawdust also reduced the tensile strength of the composites. Therefore, the sawdust reinforced epoxy resin composites are more brittle and stiff than the neat epoxy resins.

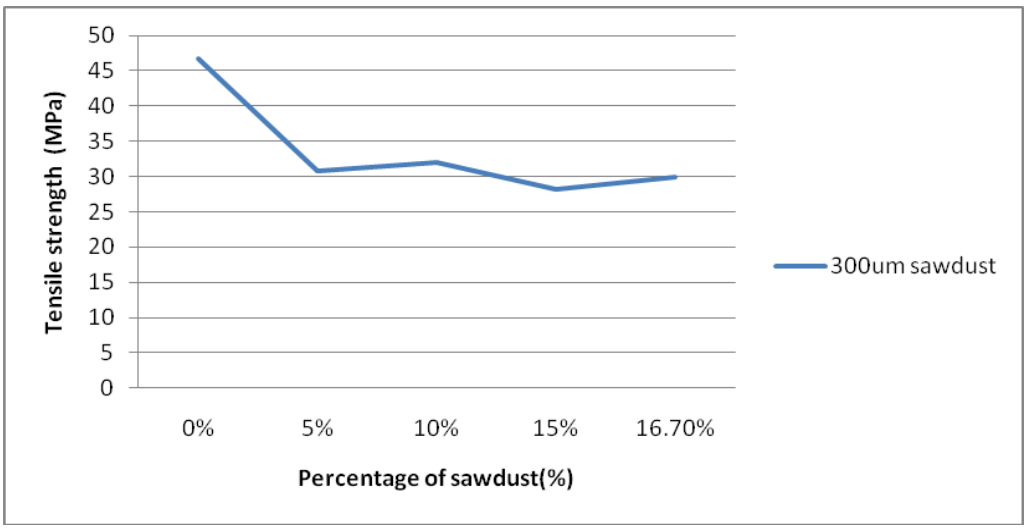


Figure 4.35: Tensile strength versus percentage of 300 µm sawdust

Figure 4.36 illustrates the 0.05% offset yield strength of the samples filled with 300 µm sawdust. It was found that the highest yield strength was 21.615 MPa at 0% of sawdust. The value of the yield strength then fell dramatically to 14.979 MPa at 5% of sawdust. After this point, the value of yield strength fluctuated slightly between 15.58 and 14.21 MPa. Therefore, it can be concluded that the present of sawdust as fillers reduced the yield strength of the epoxy composites. Therefore, the sawdust reinforced epoxy resin composites are more brittle than the neat epoxy resins.

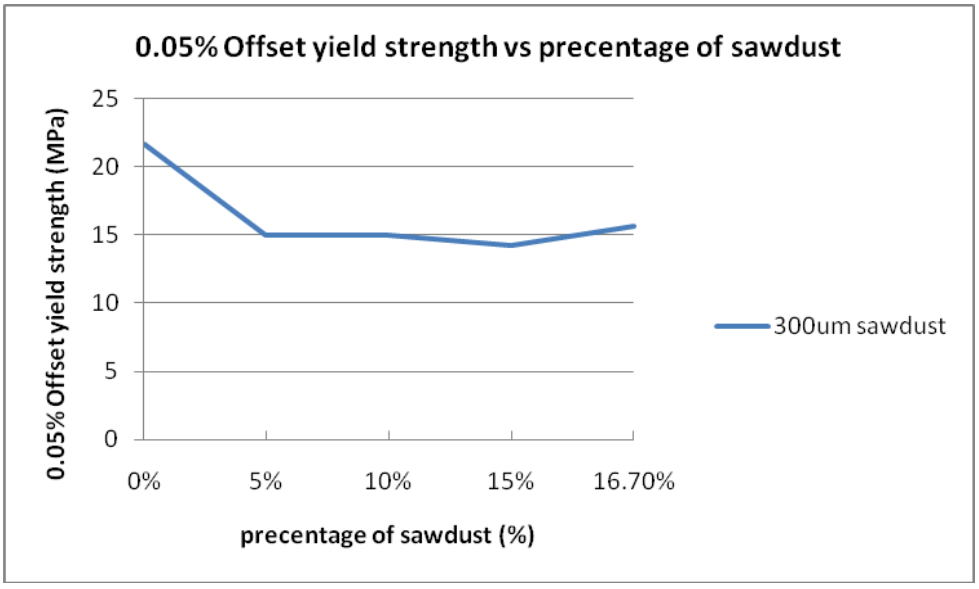


Figure 4.36: 0.05% Offset yield strength versus percentage of 300 µm sawdust

4.3.3 Test results for the composites filled with 425 µm sawdust

Figures 4.37, 4.38 and 4.39 are produced base on the processed data listed in Table 4.4. Figure 4.37 illustrates the Young's modulus of the specimens filled with the sawdust of 425 µm in diameter. The Young's modulus gradually increased from 2521 MPa to 3245 MPa as the percentage of sawdust increased from 0% to 16.70%. The Young's modulus increased 28.7% by comparing the value at 16.7% of sawdust and the value at 0% of sawdust. The trend in Figure 4.37 shows Young's modulus increases with the percentage of sawdust increases.

Table 4.4: Processed mean values of the test results for samples reinforced with 425 µm sawdust

Samples filled with 425µm sawdust					
Percentage of saw dust	0%	5%	10%	15%	16.70%
Young's modulus MPa	2521	2877	3020	3001	3245
Tensile strength MPa	46.81	11.82	29.57	28.85	28.32
0.05% offset Yield strength (MPa)	21.615	9.48	14.18	14.23	13.56

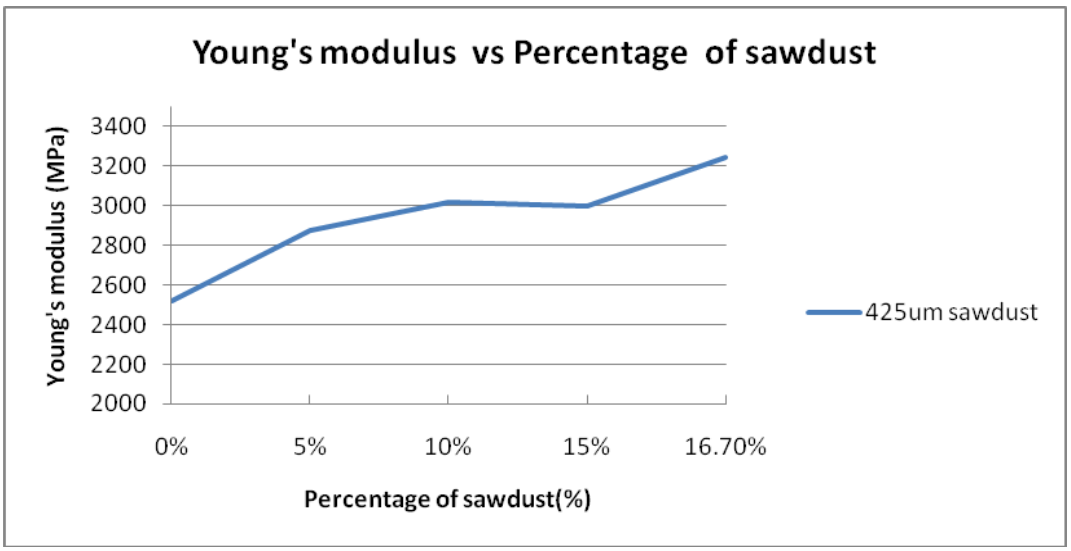


Figure 4.37: Young's modulus versus percentage of 425 µm sawdust

Figure 4.38 shows the tensile strength of specimens filled with the sawdust of 425 µm in diameter. The highest value of the tensile strength was observed from Figure 4.38 to be 46.81 MPa at 0% of sawdust. After this point, the value then declined sharply to 11.82 MPa which was the lowest value in this group of samples as the percentage of the sawdust increased from 0% to 5%. The tensile strength then bounced back to 29.57 MPa when the percentage of sawdust increased from 5% to 10%. The tensile strength reduced slightly when the percentage of sawdust increased from 10% to 16.7%. The tensile strength only dropped 4.2% since the percentage of sawdust increased from 10% to 16.7%. Hence, within the range between 10% to 16.7% of sawdust, the increase in percentage of sawdust will only reduce the tensile strength slightly. The sudden drop of tensile strength at 5% of sawdust was suspicious as it was inconsistent with the trend of the curve in Figure 4.38. The expected value at 5% of sawdust should be around 30 MPa. The unreasonably low value of tensile strength may due to the material decomposition caused by the hot spot in the microwave oven. Another possibility could be excessive air bubbles existed in the sample of 5% sawdust, this could be verified by microscope inspection.

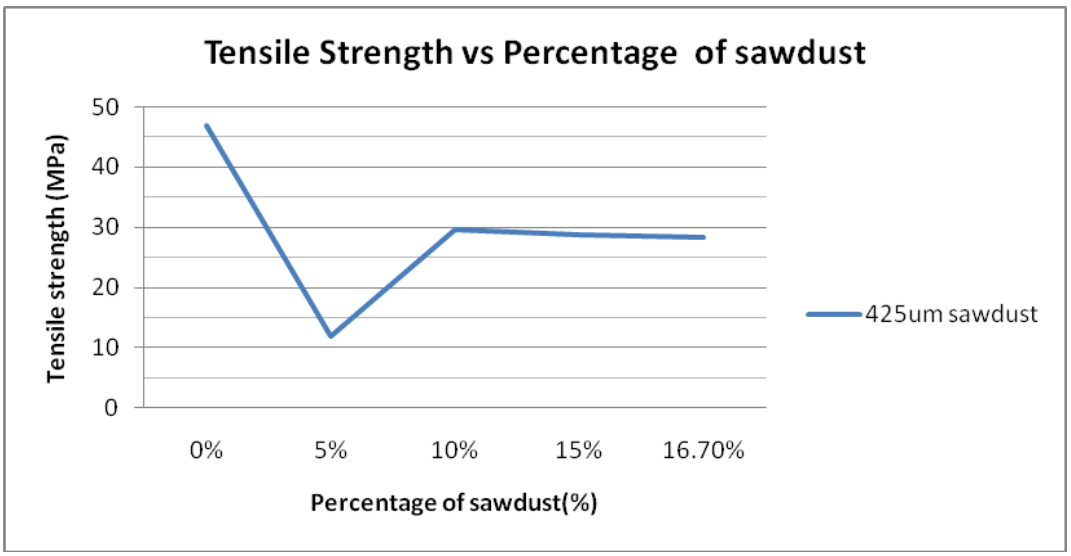


Figure 4.38: Tensile strength versus percentage of 425 µm sawdust

Figure 4.39 shows the yield strength of specimens filled with the sawdust of 425 µm in diameter. The highest value of the yield strength was observed from Figure 4.39 to be 21.615 MPa at 0% of sawdust. After this point, the value then declined sharply to 9.478 MPa which was the lowest value in this group of samples as the percentage of the sawdust increased from 0% to 5%. The yield strength then bounced back to 14.175 MPa when the percentage of sawdust increased from 5% to 10%. The yield strength reduced slightly when the percentage of sawdust increased from 10% to 16.7%. The yield strength only dropped 3.2% since the percentage of sawdust increased from 10% to 16.7%. Hence, within the range between 10% to 16.7% of sawdust, the increase in percentage of sawdust will only reduce the yield strength slightly. The sudden drop of tensile strength at 5% of sawdust was suspicious as it was inconsistent with the trend of the curve in Figure 4.39. The expected value at 5% of sawdust should be around 15 MPa. The unreasonably low value of yield strength may due to the material decomposition caused by the hot spot in the microwave oven. Another possibility could be excessive air bubbles existed in the sample of 5% sawdust, this could be verified by microscope inspection.

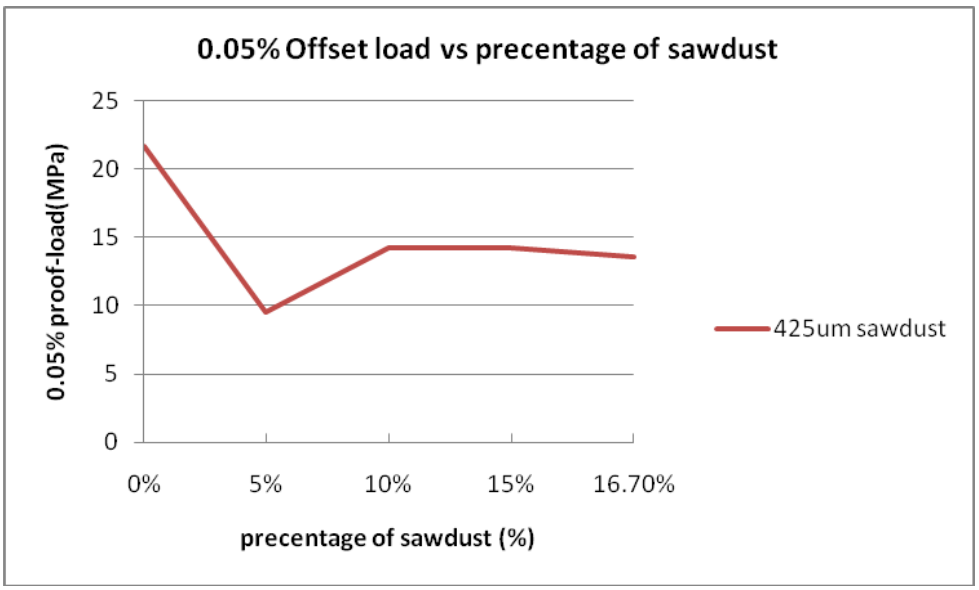


Figure 4.39: 0.05% Offset yield strength versus percentage of 425 µm sawdust

4.3.4 Test results for the composites filled with 1180 µm sawdust

Figures 4.40, 4.41 and 4.42 are produced base on the processed data listed in Table 4.5.

Table 4.5: Processed mean values of the test results for samples reinforced with 1180 µm sawdust

Samples filled with 1180 µm sawdust					
Percentage of saw dust	0%	5%	10%	15%	16.70%
Young's modulus MPa	2521	2648	2954	2843	2202
Tensile strength MPa	46.81	21.075	18.59	14.06	12.16
0.05% offset Yield strength (MPa)	21.615	9.88	11.085	8.961	8.298

Figure 4.40 shows the tensile strength of the samples filled with different percentage of 1180 µm sawdust. Initially, the pure epoxy resins had a tensile strength of 46.81 MPa which was the highest value in this group of samples. After this point, the tensile strength then rapidly dropped 55% to 21.075 MPa at 5% of sawdust. The tensile

strength then deceased gradually from 21.075 MPa to 12.16 MPa as the percentage of sawdust increased from 5% to 16.70%. Base on the trend shown in Figure 4.40, it can be concluded that the tensile strength is significantly reduced once the sawdust is added into the resins.

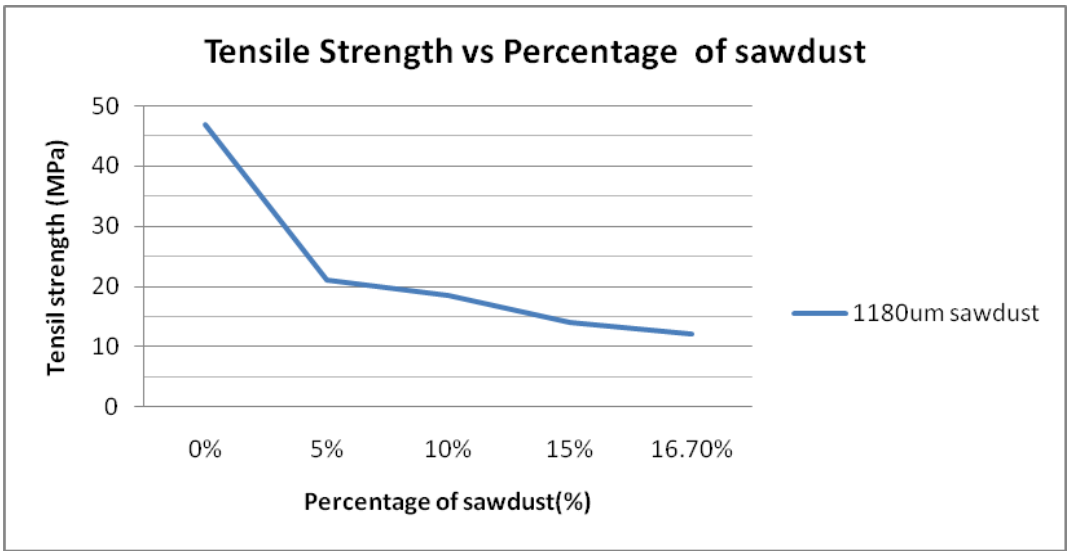


Figure 4.40: Tensile strength versus percentage of 1180 µm sawdust

Figure 4.41 shows the Young's modulus of the samples filled with different percentage of 1180 µm sawdust. The Young's modulus increased gradually from 2521 MPa to 2954 MPa as the percentage of sawdust increased from 0% to 10%. The Young's modulus then decreased slightly to 2843 MPa at 15% of sawdust. The Young's modulus fell sharply to 2202 MPa when the percentage of sawdust increased from 15% to 16.7%. The Young's modulus at 16.7% was even lower than the value of pure epoxy resin. This situation has not been observed in the other samples filled with 300 µm and 425 µm sawdust. Thus, the possible explanation for this situation could be because of the significant size of the sawdust created a lot of air gaps between the sawdust and the resins.

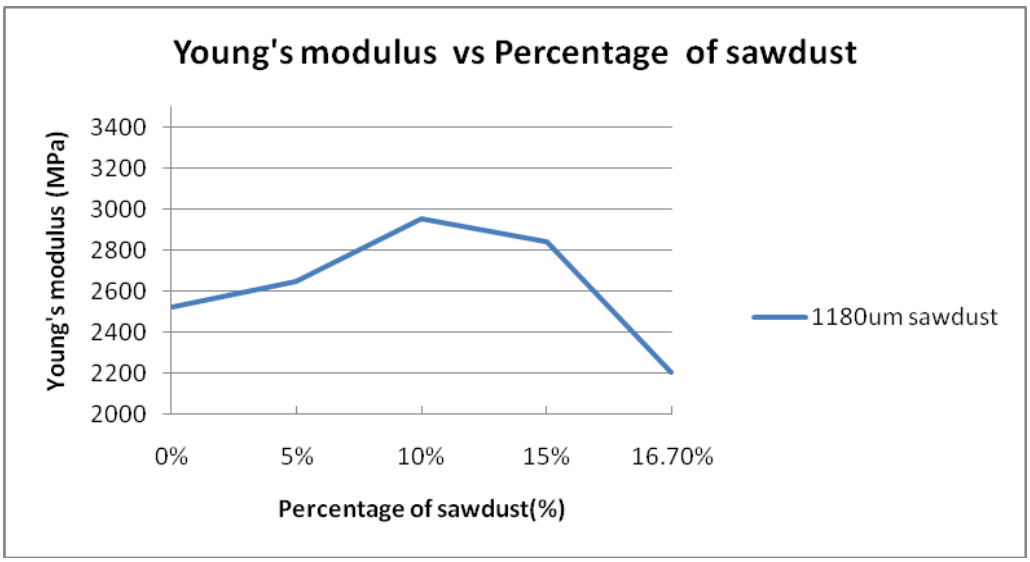


Figure 4.41: Young's modulus versus percentage of 1180 µm sawdust

Figure 4.42 shows the yield strength of the samples filled with different percentage of 1180 µm sawdust. Initially, the pure epoxy resins had yield strength of 21.615 MPa which was the highest value in this group of samples. After this point, the yield strength then rapidly dropped 54% to 9.88 MPa at 5% of sawdust. The yield strength then fluctuated slightly between 11.085 and 8.298 MPa as the percentage of sawdust increased from 10% to 16.70%. Base on the trend shown in Figure 4.42, it can be concluded that the yield strength is significantly reduced once the sawdust is added into the resins.

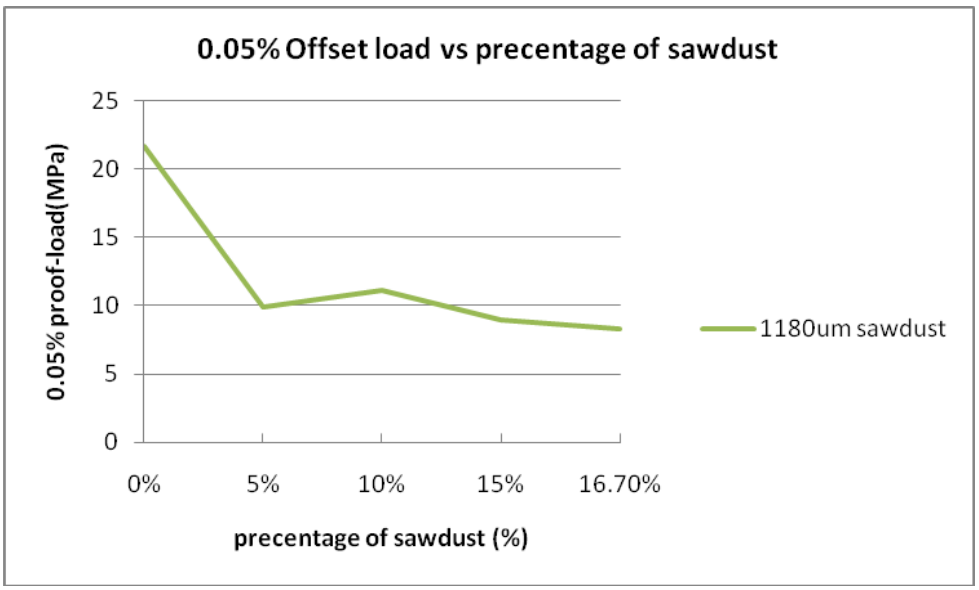


Figure 4.42: 0.05% Offset yield strength versus percentage of 1180 µm sawdust

Figure 4.43 combines Figure 4.34, Figure 4.37 and Figure 4.41 to make a comparison of Young's modulus between the samples filled with 300 µm sawdust, 425µm sawdust and 1180µm sawdust. It can be seen from Figure 4.43 the samples filled with 425 µm sawdust has the best performance for the test of Young's modulus in general. Figure 4.43 also shows the Young's modulus has been improved for the sawdust reinforced samples, compared with the pure epoxy resins, except for the samples filled with 16.7% of 1180 µm sawdust.

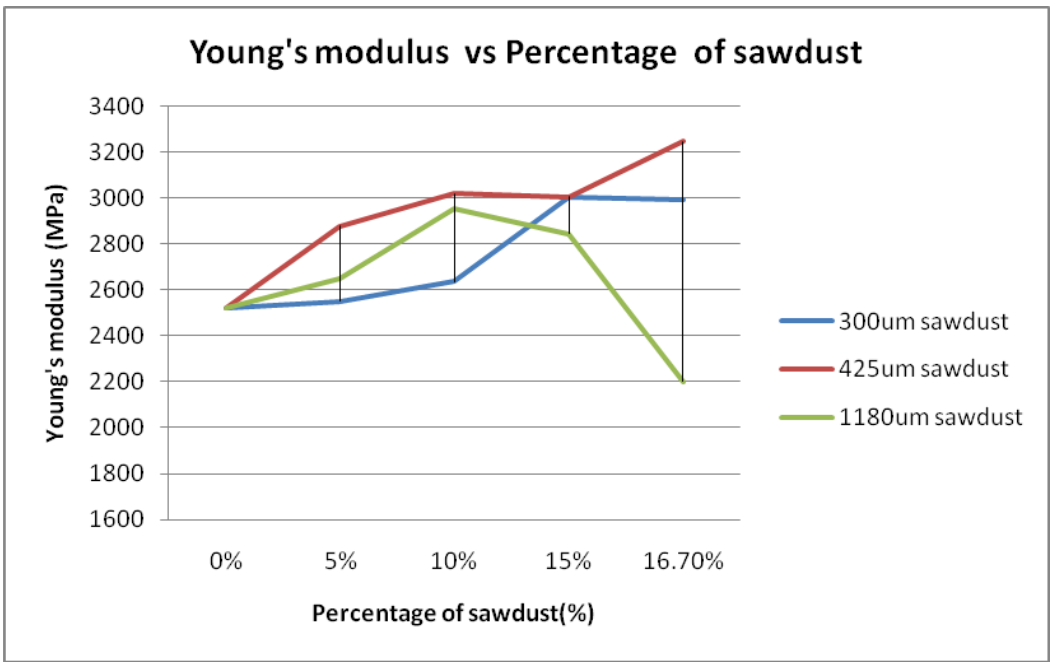


Figure 4.43: Comparison of Young's modulus for 300 µm, 425 µm and 1180 µm sawdust

Figure 4.44 provides a comparison for the tensile strength between the samples filled with 300 µm, 425 µm and 1180 µm sawdust. Despite the extremely low value of tensile strength for the possible defective samples filled with 5% of 425 µm sawdust, the samples which filled with 300 µm and 425 µm sawdust generally display better performance than the samples filled with 1180 µm sawdust.

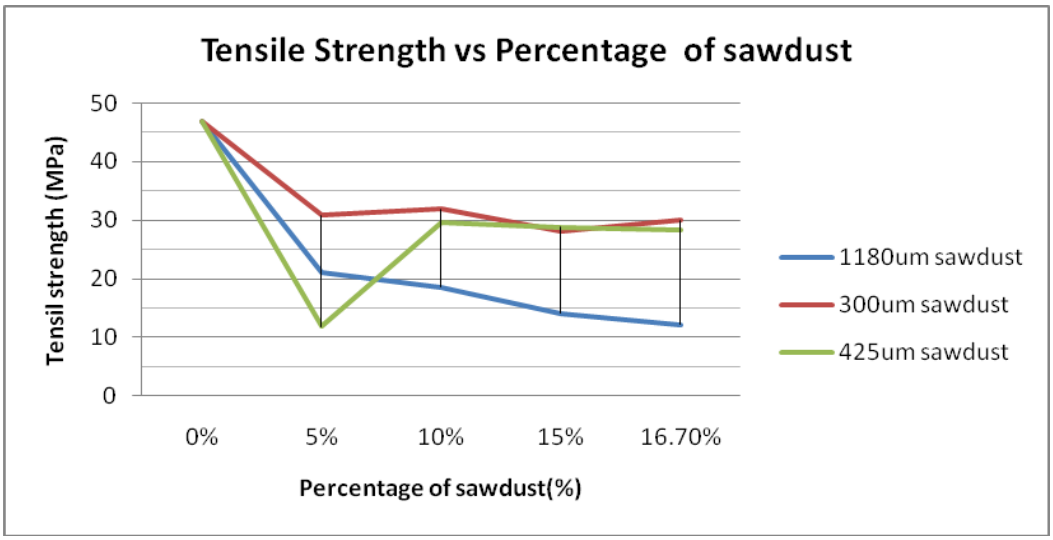


Figure 4.44: Comparison of Young's modulus for 300 µm, 425 µm and 1180 µm sawdust

Figure 4.45 provides a comparison for the yield strength between the samples filled with 300 µm, 425 µm and 1180 µm sawdust. Despite the extremely low value of yield strength for the possible defective samples filled with 5% of 425 µm sawdust, the samples which filled with 300 µm and 425 µm sawdust generally display better performance in yield strength than the samples filled with 1180 µm sawdust.

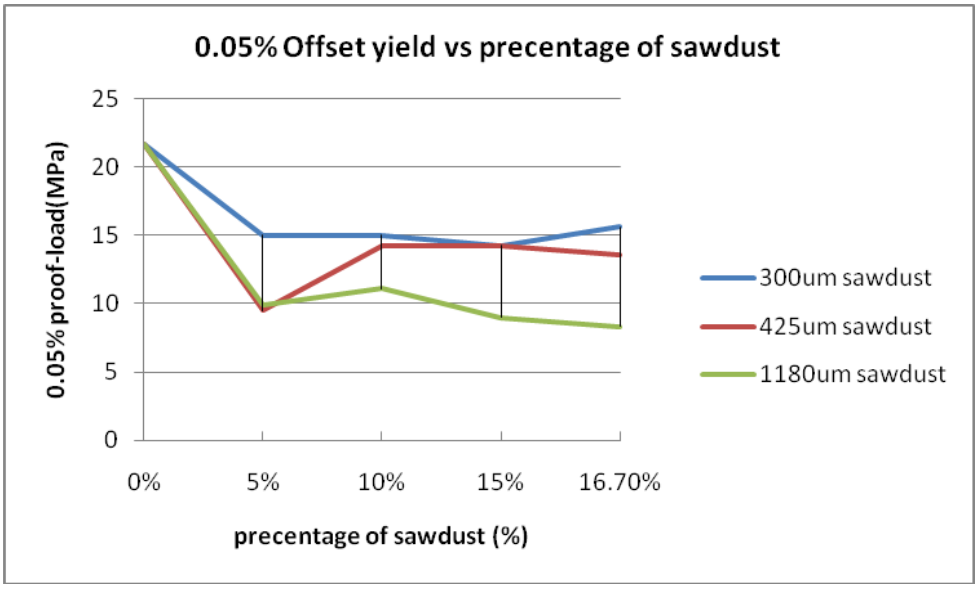


Figure 4.45: Comparison of 0.05% offset yield strength for 300 µm, 425 µm and 1180 µm sawdust

4.4 Microscope inspection results

In order to easily compare the results, all of the pictures were magnified at the same scale of 67 times of the real objects. Figures 4.46, 4.47, 4.48 and 4.49 are the microscope pictures for the samples reinforced with 5% 425 µm sawdust at a magnification of 67 times. Figures 4.50 and 4.51 show the microscope images for the samples reinforced with 10% and 15% 425 µm sawdust respectively. By comparing Figures 4.46, 4.47, 4.48 and 4.49 with Figures 4.50 and 4.51, it can be found that the samples reinforced with 5% 425 µm are generally have larger air bubbles than the samples reinforced with 10% and 15% 425 µm sawdust. It is clear now that the considerably large air bubbles should be the main cause for the unexpected low value of tensile strength and yield strength of the samples reinforced 5% 425 µm sawdust.

Hence, it is important to ensure the samples are well-mixed, so that the presents of air bubbles inside the samples are minimized.

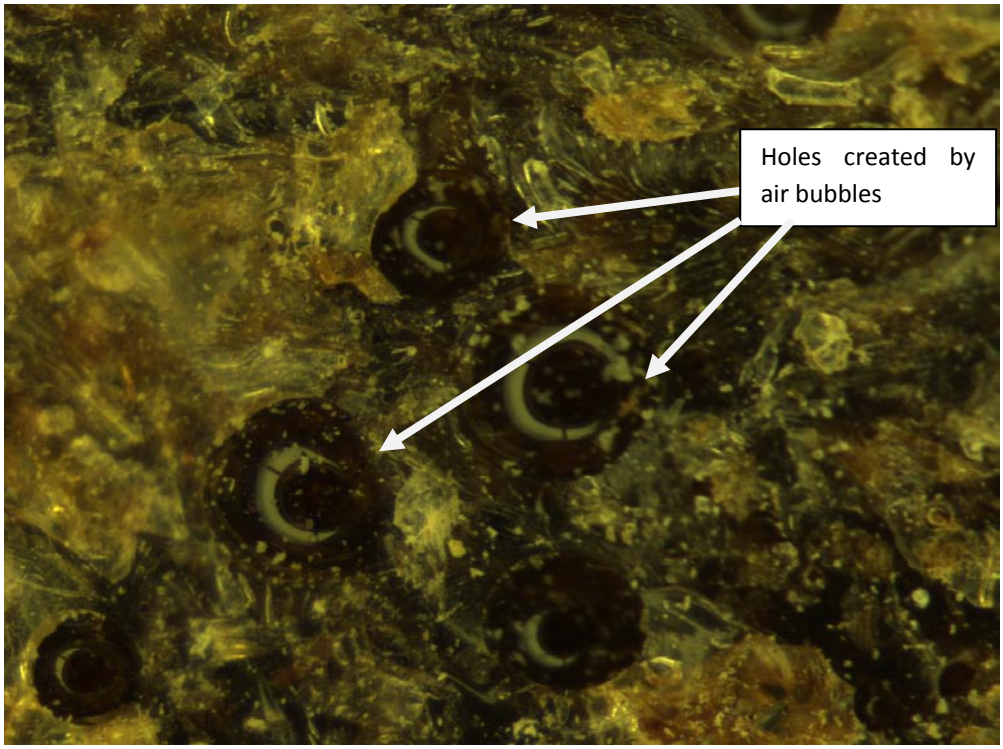


Figure 4.46: sample 1 of the epoxy resins reinforces with 5% 425 µm sawdust at a magnification of 67 times

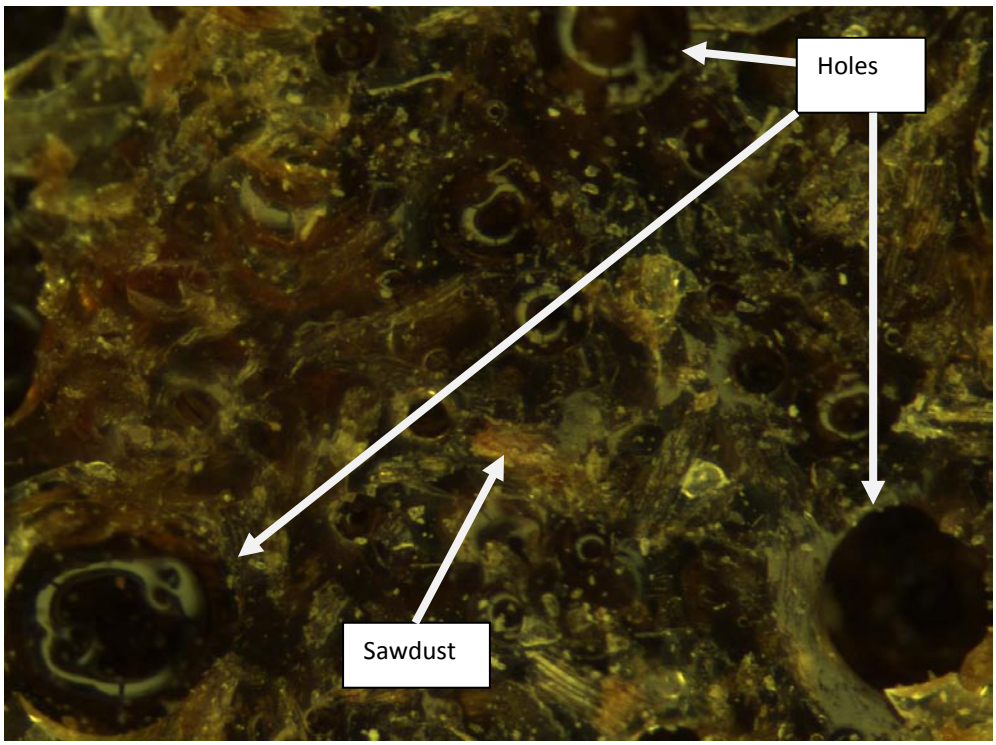


Figure 4.47: sample 2 of the epoxy resins reinforces with 5% 425 µm sawdust at a magnification of 67 times

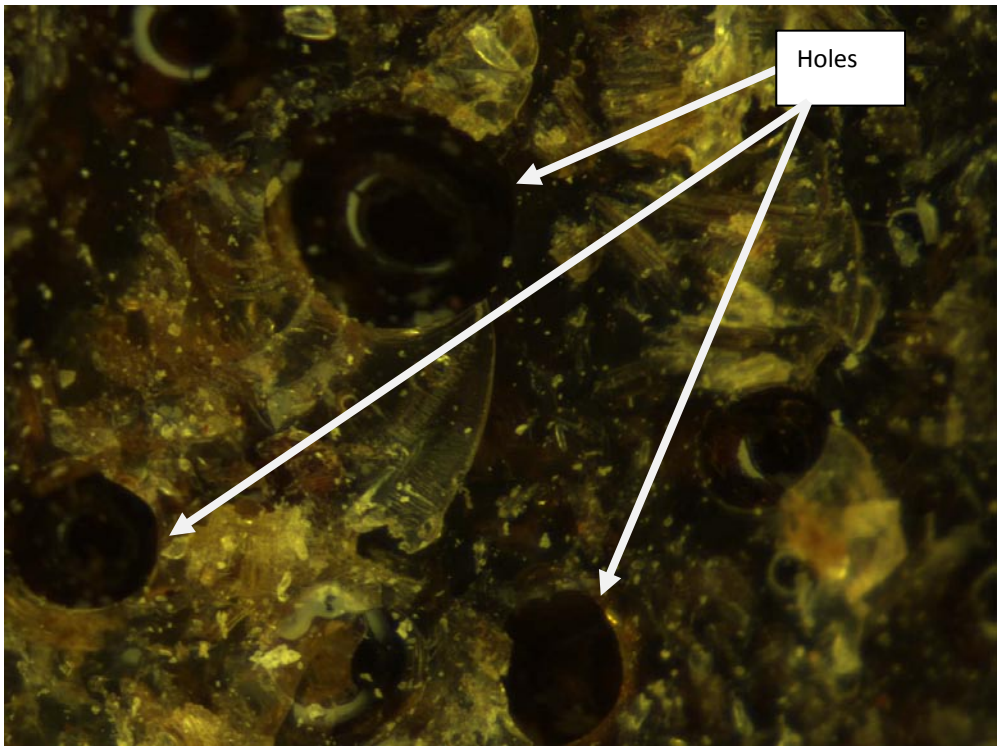


Figure 4.48: sample 3 of the epoxy resins reinforces with 5% 425 µm sawdust at a magnification of 67 times

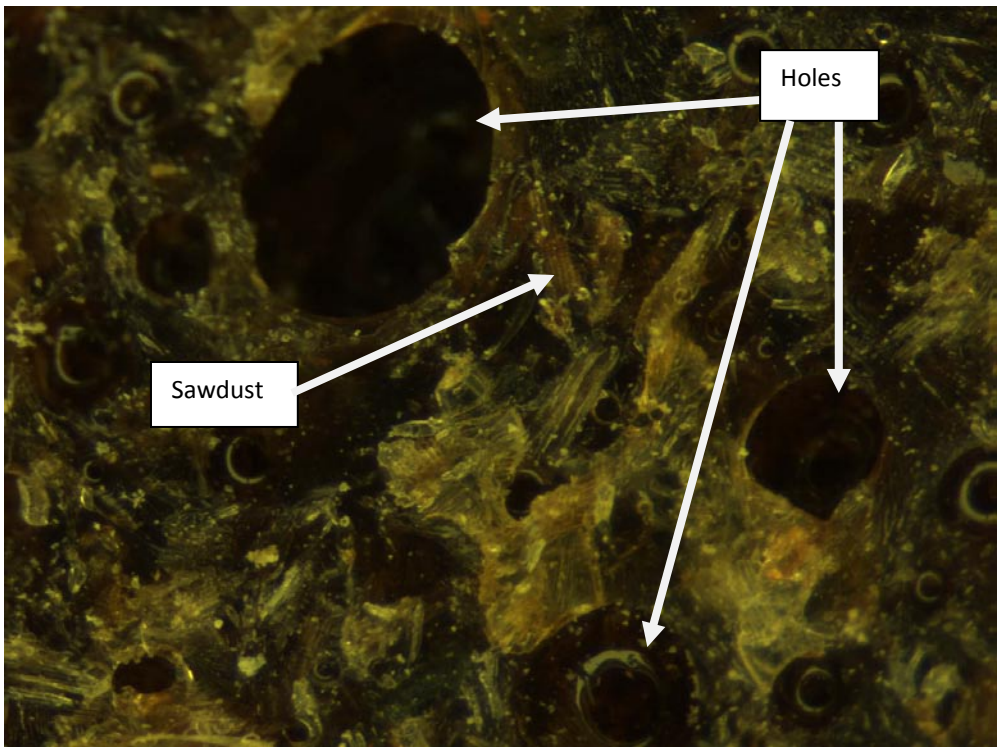


Figure 4.49: sample 4 of the epoxy resins reinforces with 5% 425 µm sawdust at a magnification of 67 times

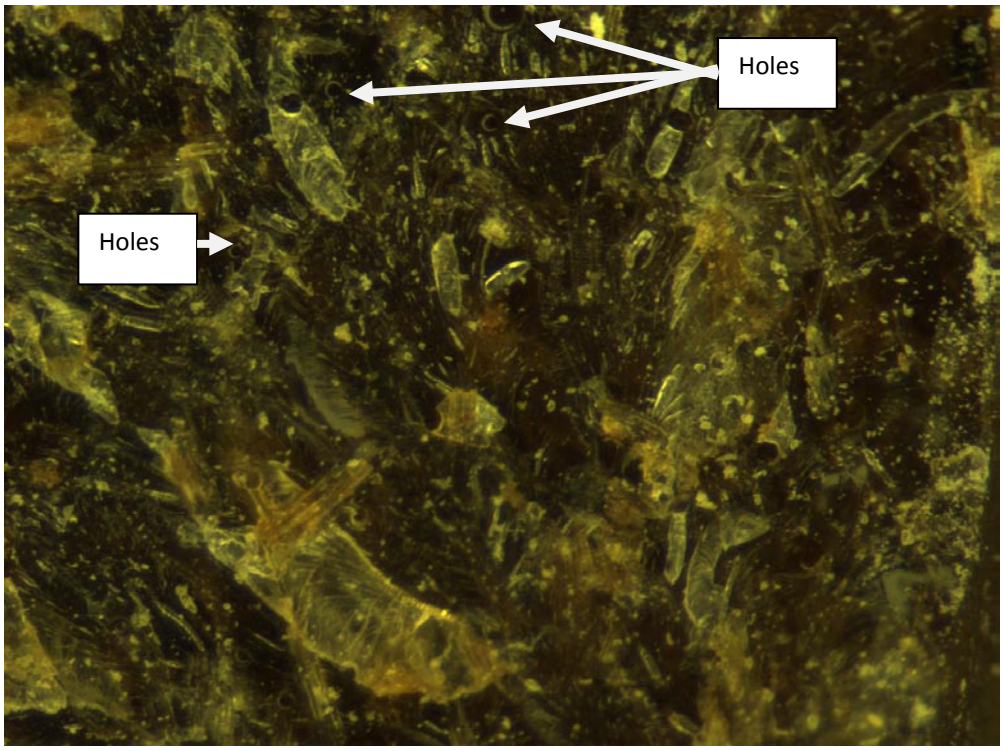


Figure 4.50: sample 2 of the epoxy resins reinforces with 10% 425 µm sawdust at a magnification of 67 times

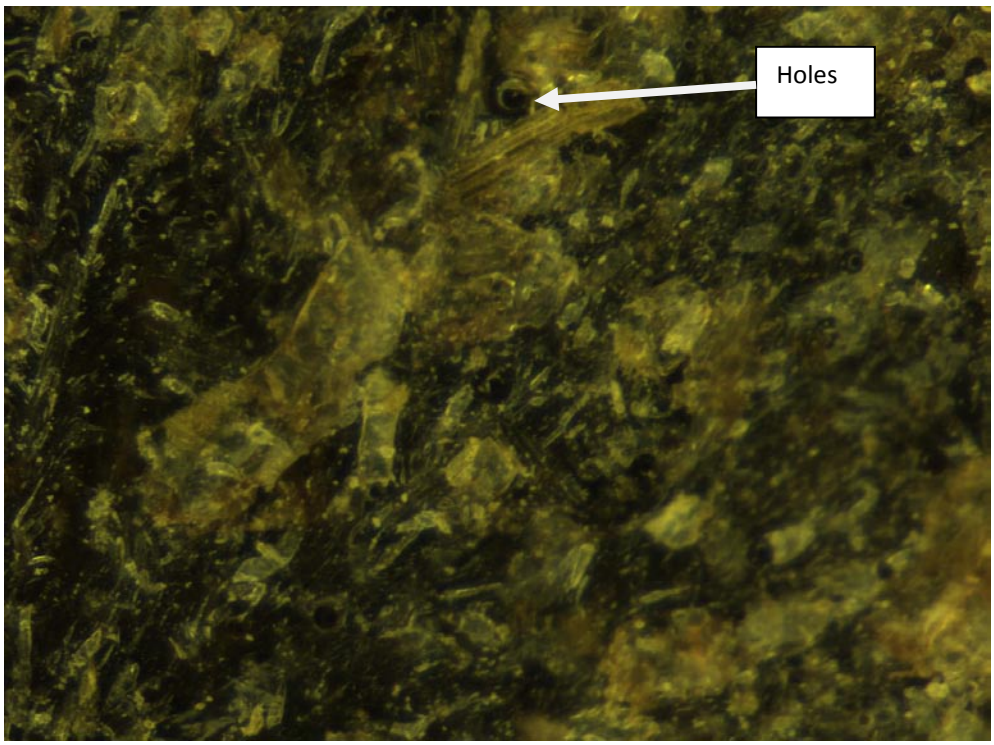


Figure 4.51: sample 1 of the epoxy resins reinforces with 15% 425 µm sawdust at a magnification of 67 times

Figure 4.52 shows the micrograph for the sample reinforced with 10% 300 µm sawdust. Figures 4.53 and 4.54 show the micrographs for the samples reinforced with 16.7% and 15% sawdust respectively. By comparing Figure 4.52 with Figures 4.53 and 4.54, it can be found that the larger the sawdust are, the larger the air bubble will be introduced in the samples. Hence, the epoxy resins should be mixed up with finer sawdust to minimise the size of the air bubbles.

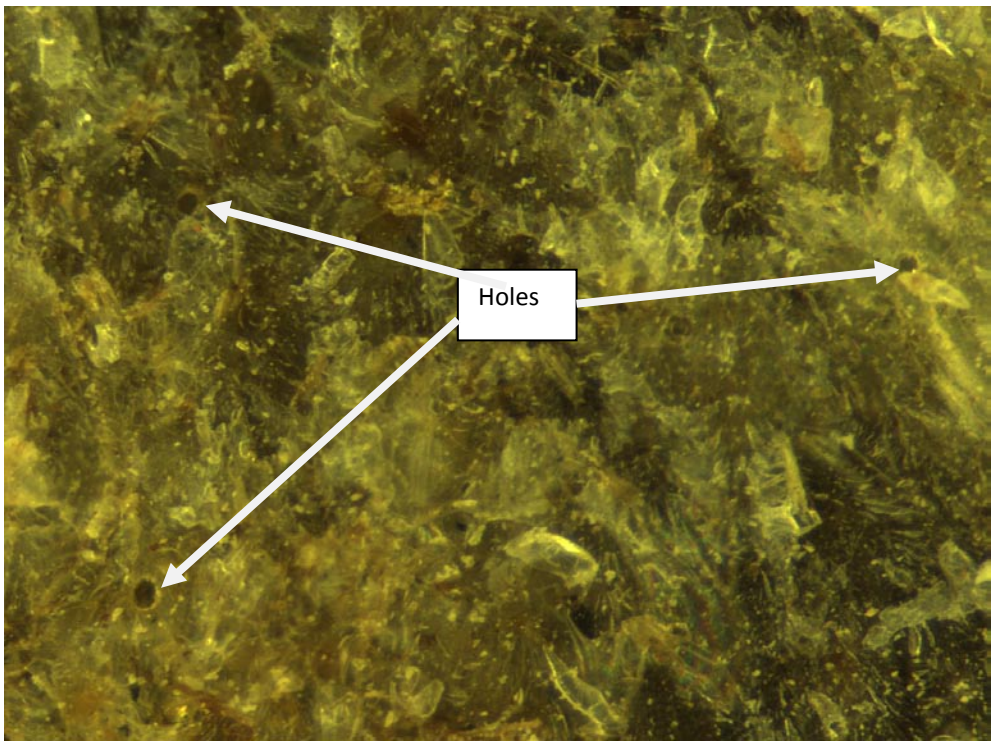


Figure 4.52: sample 1 of the epoxy resins reinforces with 10% 300 µm sawdust at a magnification of 67 times

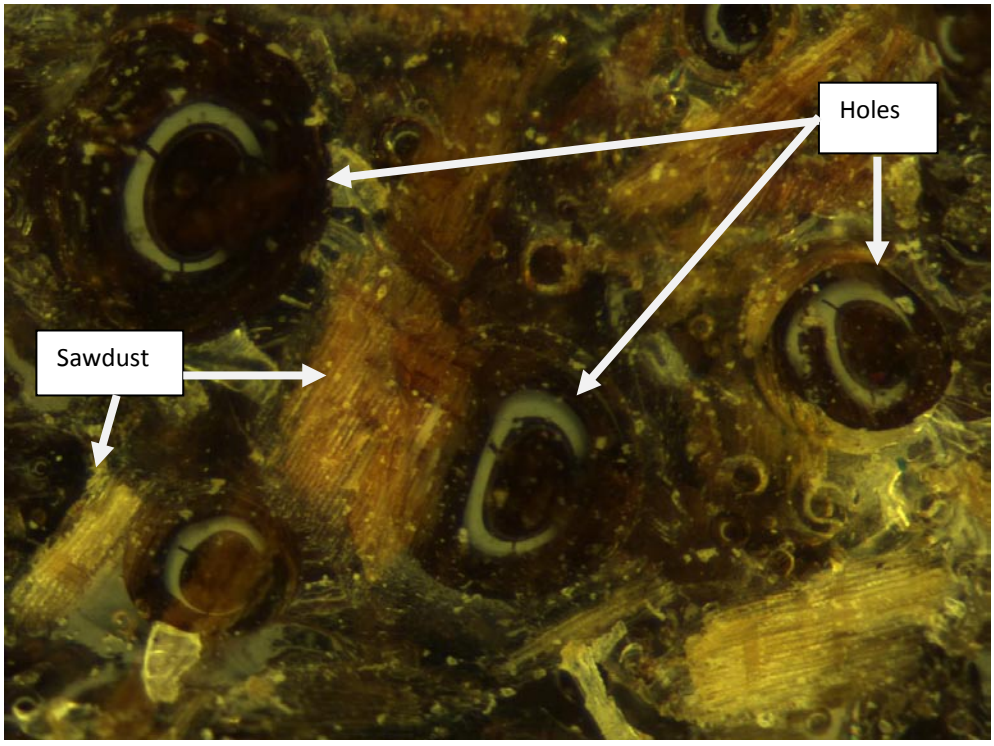


Figure 4.53: sample 3 of the epoxy resins reinforces with 16.7% 1180 µm sawdust at a magnification of 67 times

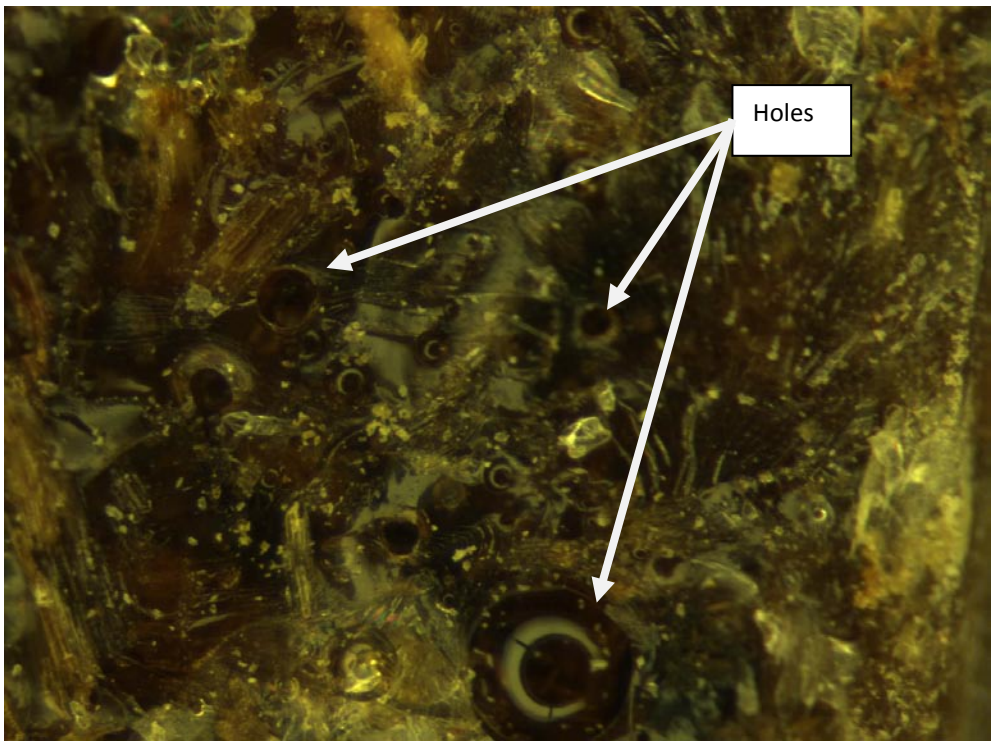


Figure 4.54: sample 1 of the epoxy resins reinforces with 15% 1180 µm sawdust at a magnification of 67 times

4.5 Cost Analysis

The cost of the resin was $ 17.3 per kg, whereas the cost of the harder was 35.14 per kg. The sawdust was $ 2296 per ton which is equal to $2.3 per kg. Since the samples reinforced with 16.7% 425 µm sawdust had the highest value of Young's modulus, they are used as examples to perform the cost analysis. The cost for making 1 kg of the composite which reinforce with 16.7% sawdust = 0.666 x $ 17.3 + 0.167 x $35.14 + 0.167 x $2.3 = $ 17.77. The cost for making 1 kg of the pure samples = 0.8 x $17.3 + 0.2 x $35.14 = $20.87. By comparing the samples reinforced with 16.7% 425 µm sawdust with pure epoxy resin samples, the reduction in cost is about 14.85%; whereas the increase in Young's modulus = (3245- 2521)/3245 = 22.3%. However, the reduction in tensile strength = (46.81- 28.32) / 46.81 = 39.5 %, and the reduction in 0.05% offset yield strength = (21.62 - 13.56) / 21.62 = 37.3%. It can be concluded that the addition of sawdust reduced the cost for the epoxy composites and improved the Young's modulus of the epoxy composites, but on the other hand, the addition of sawdust also reduced the tensile strength and yield strength.

4.6 Industrial applications

Epoxy resins have been used as substrates for making Printed Circuit Boards (PCB). The electronic circuits mount on PCBs transmit signals in microwaves. If the materials used for making PCBs tend to absorb a large portion of energy from microwaves that would not only attenuate the signal intensity, but also would generate more thermal noise as the temperature rises. Furthermore, PCBs should be able to withstand high operation temperature and the thermal strain of soldering process and still retain a reasonable physical strength. Hence materials which are used for making PCB substrates are required to have low dielectric constant, low loss tangent and high glass transition temperature. The typical required values for the dielectric constant and the loss tangent should be less or equal to 3 and 0.002 respectively. This report has found that oven cured epoxy resins have the lowest dielectric constant, the lowest loss tangent, and the highest glass transition temperature. Hence, oven cured epoxy resin is the best candidate for making PCBs. Although the addition of sawdust has no effect on loss tangent and glass transition temperature, this report still recommends using sawdust reinforced epoxy resin for economical reasons.

In material processing industry, it often requires the materials have high loss tangent, so that the materials can be heat up by microwave more efficiently. The loss tangents of the sawdust reinforced epoxy resins were slightly higher than the loss tangents of pour epoxy resins. Hence, the sawdust reinforced epoxy resin is slightly more efficient to be post-cured in microwave than pure epoxy resin.

5. CONCLUSIONS

5.1 Introduction

This project has evaluated the dielectric, thermal and mechanical properties of epoxy resins reinforced with varying percentage of sawdust. This section will summaries the important findings throughout the project.

5.2 Conclusions

It has been found that the maximum percentage of sawdust can mix with epoxy resins are 16.7%, any more sawdust added into the composites would be too stick to mix with.

5.2.1 Loss tangent and Dielectric constant measurement

This study has found that the oven cured samples had the lowest loss tangent and microwave cured samples had the highest loss tangent. The loss tangents of the sawdust reinforced epoxy resins were slightly higher than the loss tangents of pour epoxy resins. Hence, the sawdust reinforced epoxy resin is slightly more efficient to be post-cured in microwave than pure epoxy resin. Moreover, the study also found that the samples reinforced with 15% sawdust have the highest dielectric constant. The samples reinforced with 0% sawdust had the lowest dielectric constant. Oven cured samples had the lowest dielectric constant and naturally cured samples had the highest dielectric constant.

5.2.2 DMA test

This study has found the oven cured samples had the highest glass transition temperature and naturally cured samples had the lowest glass transition temperature. The study also found the addition of sawdust did not have significant effects on glass transition temperature.

5.2.3 Tensile test

The samples filled with 425 μm sawdust had the best performance for the test of Young's modulus in general. This study discovered that the Young's modulus had been improved for the sawdust reinforced samples compare with the pure epoxy resins. However, the addition of the sawdust reduced the tensile strength and yield strength of the epoxy resin composites. Furthermore, the test also found the samples which filled with 300 μm and 425 μm sawdust generally displayed better performance for tensile strength than the samples filled with 1180 μm sawdust. This phenomenon may due to the large size of sawdust induced larger size of air bubbles in the samples.

5.2.4 Microscope inspection

The inspection has found that the samples reinforced with 5% 425 μm sawdust which had unreasonable low value of tensile strength and yield strength generally had considerably large size of air bubbles. This observation verified that the presence of large size of air bubbles in the samples was the main reason for the weakening of tensile strength and yield strength of the samples. It also found that the larger size of sawdust tend to induce larger size of air bubbles in the samples. Hence, the epoxy resins should be mixed up with finer sawdust to minimise the size of the air bubbles in the samples.

5.2.5 Industrial applications

This report has found that oven cured epoxy resins have the lowest dielectric constant, the lowest loss tangent, and the highest glass transition temperature. Hence, oven cured epoxy resin is the best candidate for making PCBs. The loss tangents of the sawdust reinforced epoxy resins were slightly higher than the loss tangents of pour epoxy resins. Hence, the sawdust reinforced epoxy resin is slightly more efficient to be post-cured in microwave than pure epoxy resin.

5.3 Further Research and Recommendations

The LCR meter used in the dielectric constant and loss tangent measurement can only measure the dielectric constant and the loss tangent up to 100 kHz. However, most frequencies of the microwaves used in both material processing industry and electronic industry are above 2 GHz. Furthermore, the results obtained from the parallel plate measurement method would contain inherent errors due to the air gap effect and the edge effect. Hence, it is necessary to measure the dielectric constant and loss tangent with better equipment to provide more accurate results for practical applications.

APPENDIX A

A.1 – Project Specification

University of Southern Queensland

FACULTY OF ENGINEERING AND SURVEYING

ENG4111/4112 Research Project
PROJECT SPECIFICATION

FOR:	**PING TAI**
TOPIC:	Tensile properties of sawdust reinforced epoxy composites post-cured in microwaves
SUPERVISOR:	Dr. Harry Ku, and Dr. Francisco Cardona, USQ
ENROLMENT:	ENG 4111 –S1,O,2010; ENG 4111 –S2,O,2010
PROJECT AIM:	The aim of this project is to produce a range of epoxy resin with different percentage by weight of sawdust as filler. The result of this project is to demonstrate the effect on tensile strength of the final product by adding different percentage of sawdust and the cost reduction by using cheaper filler such as sawdust.
SPONSORSHIP:	None
PROGRAMME:	**Issue B, 27nd April 2010**

11. Research the background information relating to epoxy resin, microwaves.
12. Produce six pieces of specimens with different percentage by weight of sawdust, post-cure the specimens with microwaves.
13. Implement tensile test on the specimens
14. Carry out DMA test
15. Carry out the permittivity test
16. Analyse the test results.
17. Consider the material costs and the test results to recommend the best specimens for different industrial applications.
18. Identify the possible defects on the specimens.
19. Suggest possible solution to minimise defects.

AGREED:

_____ (Student) _____

(Supervisor)

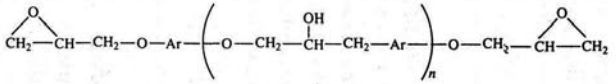
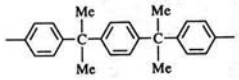
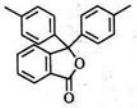
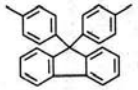
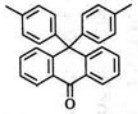
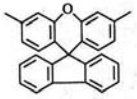
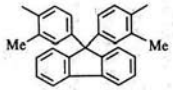
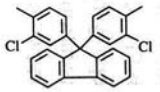
Date: / /2010 Date:27 /04/2010

Examiner/Co-examiner:_____Date: / /2010

APPENDIX B

B.1 High performance epoxy resins

Table 8.1 The Basic Properties of High-Performance Epoxy Resins

Resin –Ar–		n	Equivalent weight (g/epoxide)	Resin T_g (°C)	Melting point (°C)	Color
DGEIB[a]			240–255	1195	cP at 30°C	White clear
DGEPP		0.24	260	30		Yellow
DGEBF		0.36	305	50	132	White
DGEA		0.45	343	48	163	White
DGEFX		0.87	416	58	—	Yellowish brown
DGEBF –DiMe		0.06	257		85–95	
DGEBF –DiCl		0.02	270		198	

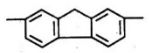
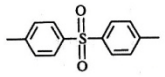
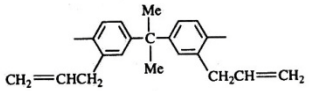
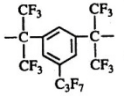
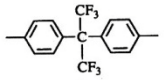
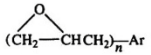
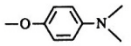
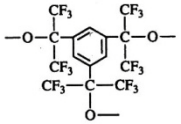
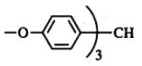
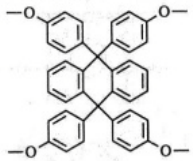
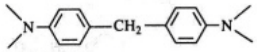
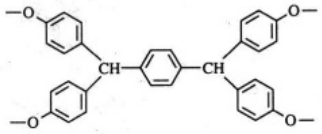
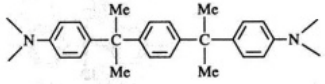
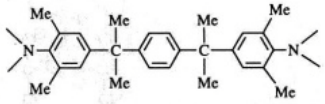
DGEFD		0.00	202	125	135–145	
DGEBS						
DGEBA –Allyl		N.A.				
DGE—GF —7F		N.A.				
DGEBA —6F		N.A.				
						
TGIC		3	103 101–111		100–104 85–110	White White
TGAP		3	110 95–107	3000 550–850	cP at 25°C cP at 25°C	
		3			56–58	White
TGEPM		3	162 220	55 85	— —	

Table 8.1 *Continued*

Resin –Ar–		*n*	Equivalent weight (g/epoxide)	Resin T_g (°C)	Melting point (°C)	Color
TGETA		4	204	143	—	White
TGDDM		4	117–134 110	8000–18000 3000–6000	cP at 50°C cP at 50°C	
						
TGBAP		4	150–170	23	50	
TGMBAP		4	185–205	41	65	

APPENDIX C

C.1 Tensile test results

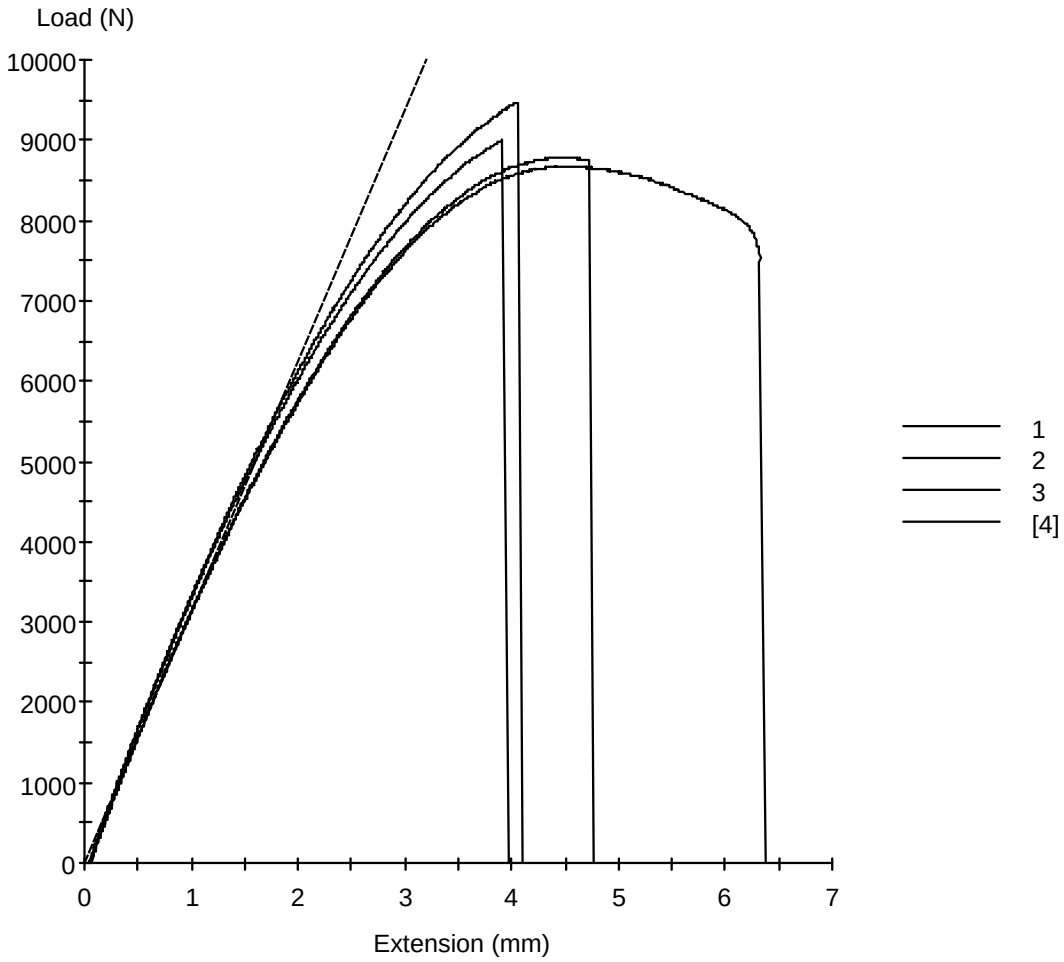
Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm
0% sawdust

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.260	25.530	185	8675	46.81	7543	40.70
2	7.820	25.000	196	9472	48.45	9472	48.45
3	7.700	25.050	193	8997	46.64	8997	46.64
4	7.470	25.100	187	8785	46.86	8752	46.68
Mean	**7.562**	**25.170**	**190**	**8982**	**47.19**	**8691**	**45.62**
Std Dev	**0.249**	**0.243**	**5**	**353**	**0.85**	**822**	**3.39**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	6.324	23.115	4284.249	2494			
2	4.059	21.135	4131.840	2547			
3	3.904	20.711	3994.873	2550			
4	4.731	21.500	4031.129	2494			
Mean	**4.755**	**21.615**	**4110.523**	**2521**			
Std Dev	**1.106**	**1.050**	**129.504**	**31**			


Load (N)
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
0
1
2
3
4
5
6
7
Extension (mm)
1
2
3
[4]

5%300um sawdust

Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

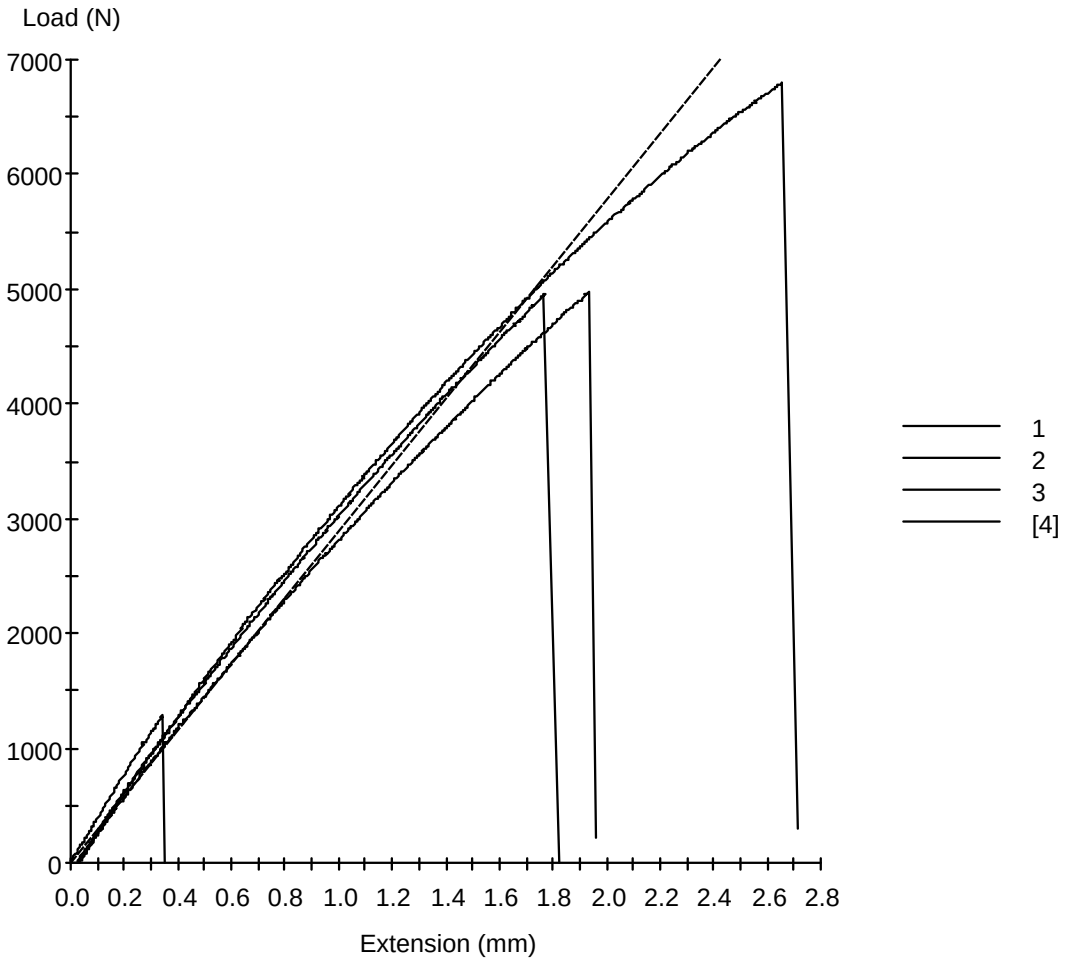
Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.150	26.880	192	1286	6.69	1286	6.69
2	7.040	26.040	183	4964	27.08	4964	27.08
3	7.100	24.990	177	6805	38.35	6805	38.35
4	7.130	24.990	178	4980	27.95	4980	27.95
Mean	**7.105**	**25.725**	**183**	**4509**	**25.02**	**4509**	**25.02**
Std Dev	**0.048**	**0.915**	**7**	**2316**	**13.25**	**2316**	**13.25**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.342	5.726	1100.436	3076			
2	1.767	13.727	2516.434	2554			
3	2.654	17.868	3170.385	2665			
4	1.939	13.343	2377.453	2431			
Mean	**1.676**	**12.666**	**2291.177**	**2681**			
Std Dev	**0.969**	**5.060**	**865.843**	**280**			

Modulus =2554+2665+2431/3 = 2550

Tensile = 27.08+38.35+27.05 = 30.83


Load (N)
7000
6000
5000
4000
3000
2000
1000
0
0.0 0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6 2.8
Extension (mm)
1
2
3
[4]

5%-425um

Report Date: 7/07/2010

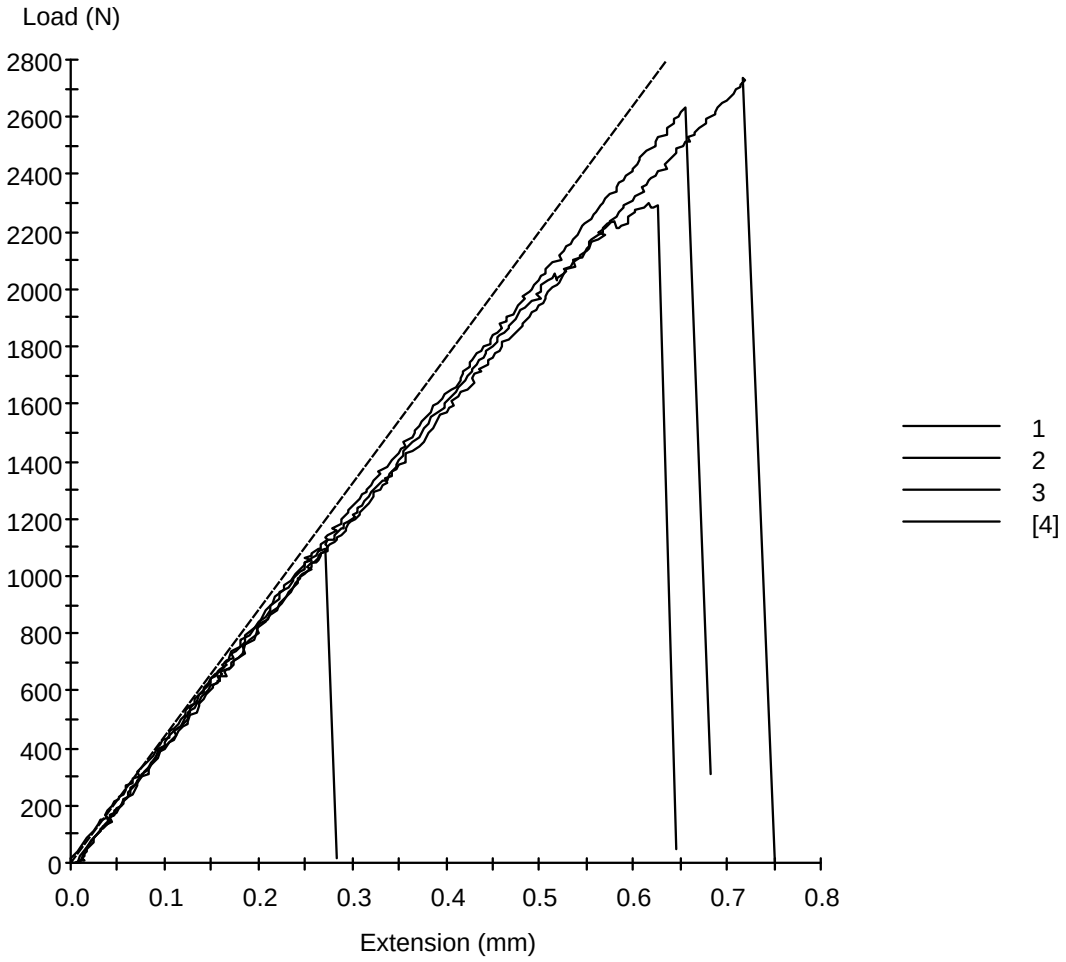
Test Date : 7/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	8.470	24.990	212	2733	12.91	2733	12.91
2	8.870	25.080	222	2301	10.34	2290	10.29
3	8.720	24.800	216	2637	12.20	2637	12.20
4	8.800	24.800	218	1094	5.01	1094	5.01
Mean	**8.715**	**24.917**	**217**	**2191**	**10.12**	**2189**	**10.10**
Std Dev	**0.174**	**0.141**	**4**	**754**	**3.57**	**754**	**3.57**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.717	9.564	2024.293	2845			
2	0.626	9.136	2032.350	2737			
3	0.655	9.733	2104.861	2893			
4	0.270	4.702	1026.078	3031			
Mean	**0.567**	**8.284**	**1796.895**	**2877**			
Std Dev	**0.201**	**2.401**	**515.154**	**122**			

Tensile = 12.91 +10.34 +12.2 /3 = 11.82


Load (N)
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
Extension (mm)
1
2
3
[4]

5% 1180 um

Report Date: 1/07/2010

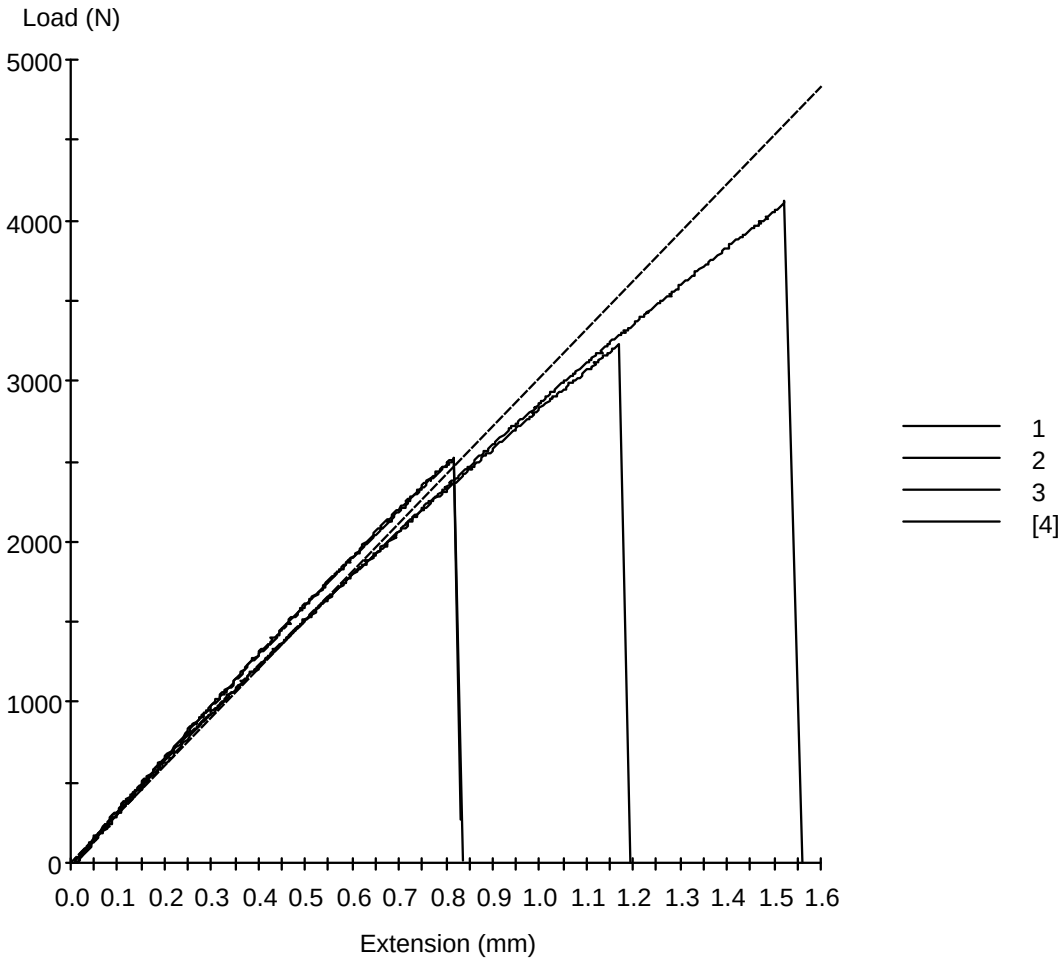
Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.790	23.870	186	2521	13.56	2521	13.56
2	7.200	24.930	179	2518	14.03	2518	14.03
3	6.910	25.140	174	3226	18.57	3226	18.57
4	6.980	25.000	174	4115	23.58	4115	23.58
Mean	**7.220**	**24.735**	**178**	**3095**	**17.44**	**3095**	**17.44**
Std Dev	**0.400**	**0.583**	**6**	**757**	**4.68**	**757**	**4.68**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.819	9.857	1832.942	2615			
2	0.819	9.463	1698.660	2732			
3	1.167	8.831	1534.165	2655			
4	1.524	11.370	1984.008	2591			
Mean	**1.082**	**9.880**	**1762.444**	**2648**			
Std Dev	**0.337**	**1.079**	**191.694**	**61**			

Tensile = 18.57 + 23.58 = 21.075


Load (N)
5000
4000
3000
2000
1000
0
0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6
Extension (mm)
1
2
3
[4]

10%300um

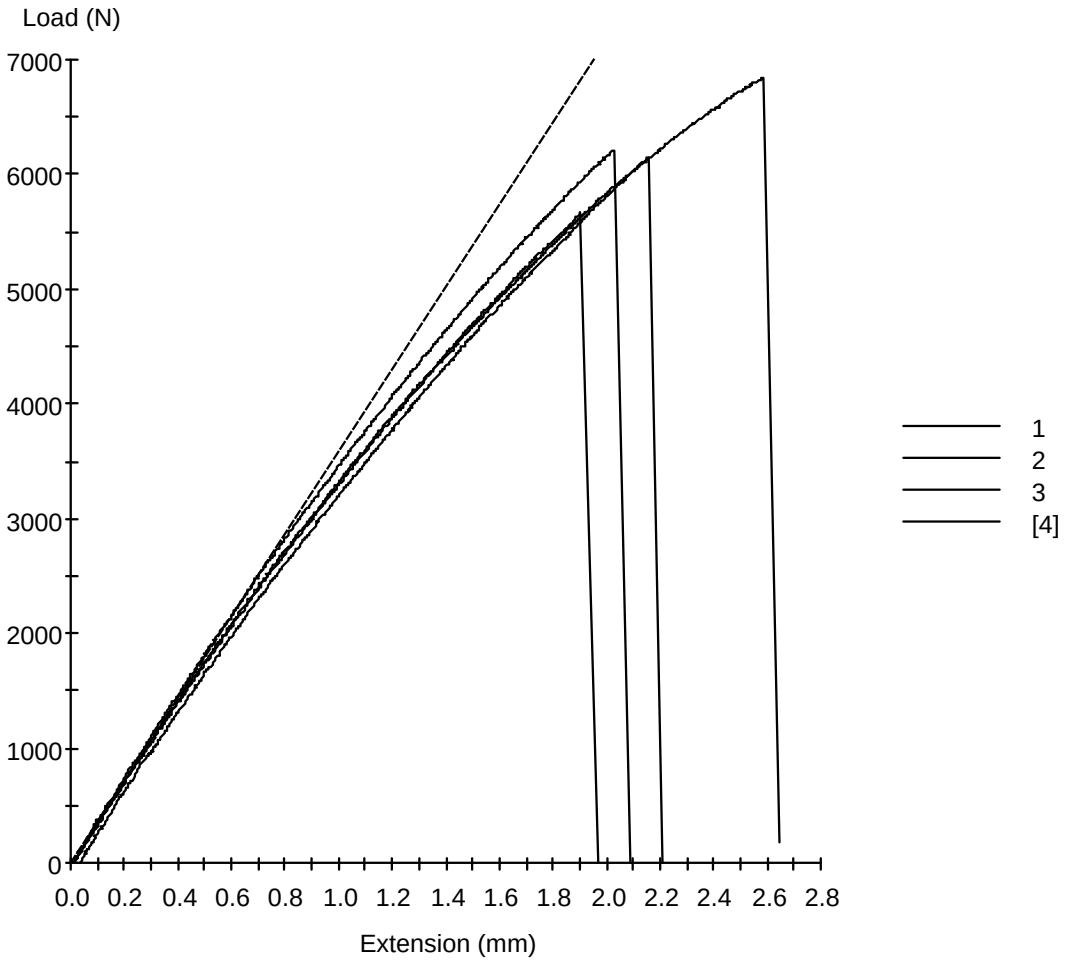
Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.680	25.210	194	6157	31.80	6157	31.80
2	7.240	27.100	196	5668	28.89	5668	28.89
3	7.520	25.250	190	6842	36.03	6842	36.03
4	7.980	25.120	200	6211	30.98	6211	30.98
Mean	**7.605**	**25.670**	**195**	**6219**	**31.93**	**6219**	**31.93**
Std Dev	**0.309**	**0.955**	**4**	**481**	**3.00**	**481**	**3.00**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	2.160	15.189	2940.763	2530			
2	1.905	13.811	2709.799	2648			
3	2.586	16.725	3175.756	2677			
4	2.027	14.309	2868.251	2687			
Mean	**2.170**	**15.008**	**2923.643**	**2635**			
Std Dev	**0.297**	**1.278**	**193.779**	**72**			


Load (N)
7000
6000
5000
4000
3000
2000
1000
0
0.0 0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6 2.8
Extension (mm)
1
2
3
[4]

10% 425um

Report Date: 7/07/2010

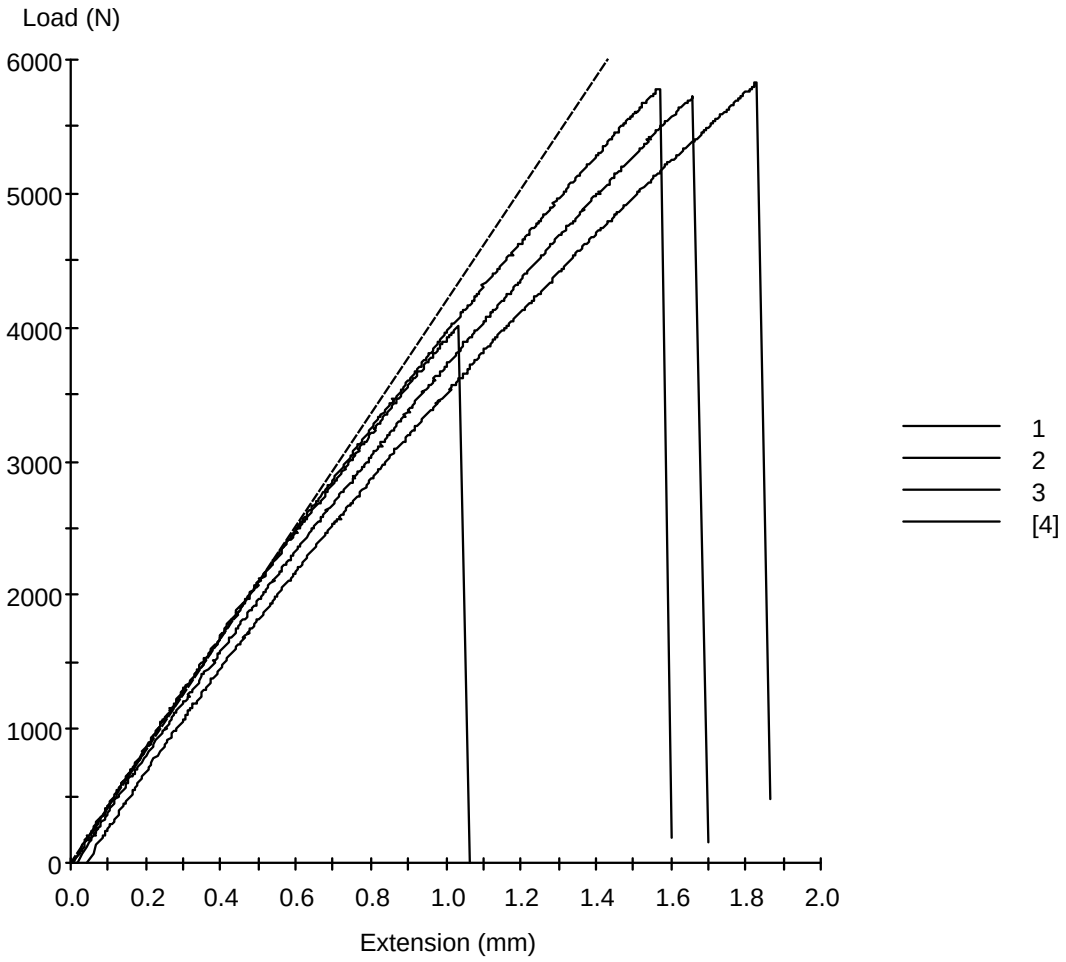
Test Date : 7/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.460	25.000	186	5831	31.26	5831	31.26
2	8.050	25.240	203	4013	19.75	4013	19.75
3	8.050	25.000	201	5730	28.47	5730	28.47
4	8.120	24.590	200	5788	28.99	5788	28.99
Mean	**7.920**	**24.958**	**198**	**5340**	**27.12**	**5340**	**27.12**
Std Dev	**0.308**	**0.270**	**8**	**886**	**5.06**	**886**	**5.06**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	1.828	16.668	3108.615	2903			
2	1.032	11.899	2417.569	3123			
3	1.657	14.106	2838.878	2903			
4	1.570	14.028	2800.943	3150			
Mean	**1.522**	**14.175**	**2791.501**	**3020**			
Std Dev	**0.343**	**1.951**	**284.441**	**135**			

Tensile = 31.26+28.47+28.99 /3 = 29.57


Load (N)
6000
5000
4000
3000
2000
1000
0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Extension (mm)
1
2
3
[4]

10%1180um

Report Date: 1/07/2010

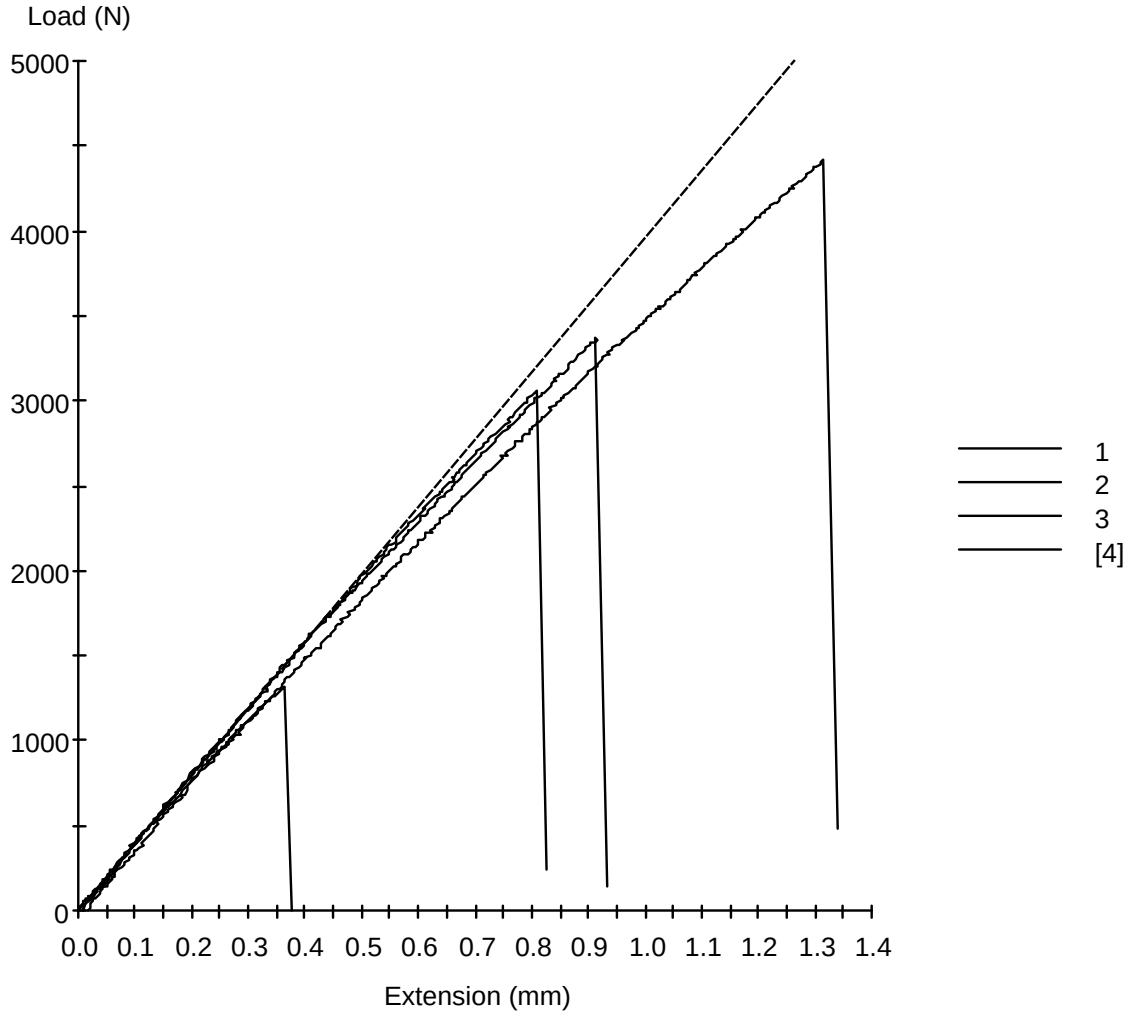
Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.640	25.880	198	3057	15.46	3057	15.46
2	7.470	25.030	187	4415	23.61	4415	23.61
3	7.670	26.350	202	3374	16.69	3374	16.69
4	7.720	26.350	203	1312	6.45	1312	6.45
Mean	**7.625**	**25.902**	**198**	**3039**	**15.55**	**3039**	**15.55**
Std Dev	**0.108**	**0.622**	**7**	**1289**	**7.05**	**1289**	**7.05**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.807	10.968	2168.645	3024			
2	1.316	11.922	2229.072	2950			
3	0.913	10.365	2094.790	2928			
4	0.366	5.482	1115.207	2915			
Mean	**0.851**	**9.684**	**1901.929**	**2954**			
Std Dev	**0.390**	**2.874**	**527.348**	**49**			

Tensile = 15.46 + 23.64 + 16.69 /3 = 18.59


Load (N)
5000
4000
3000
2000
1000
0
0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4
Extension (mm)
1
2
3
[4]

15%-300um

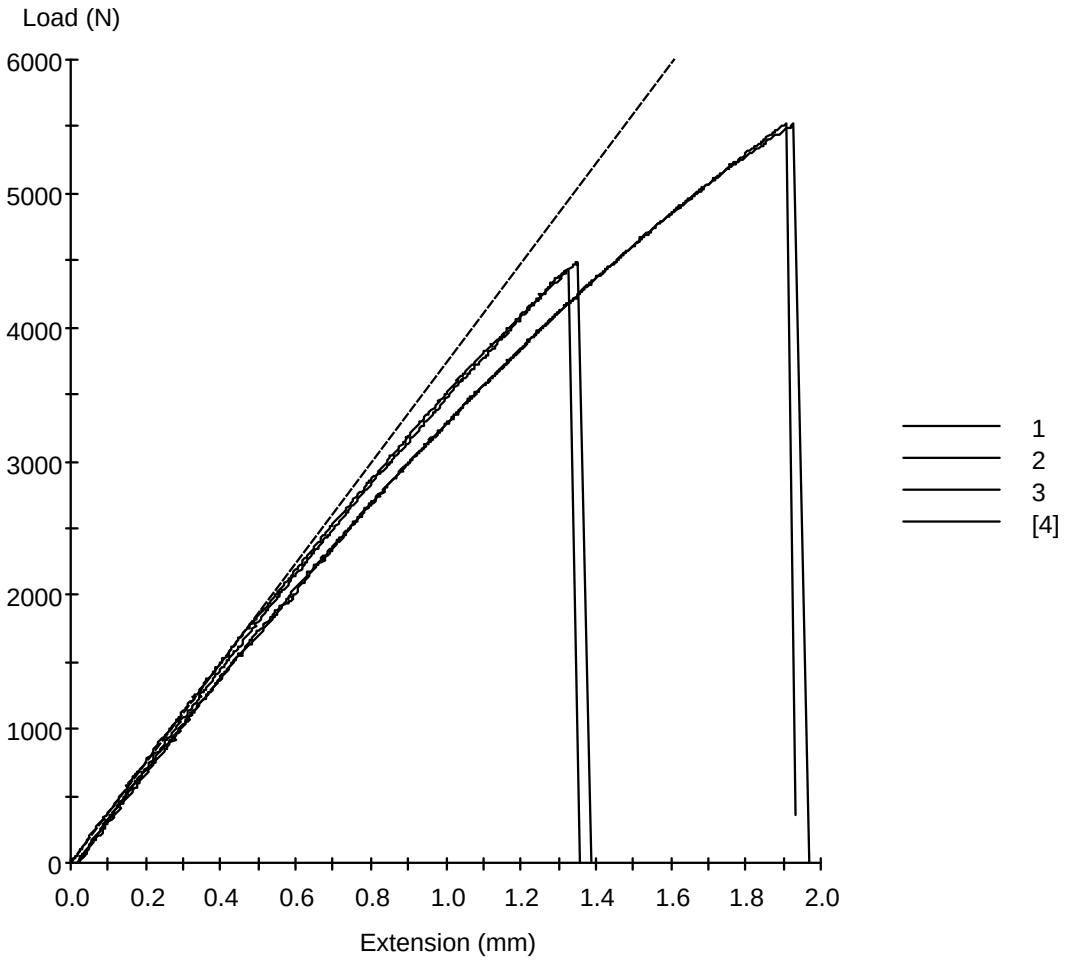
Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.000	25.060	175	5516	31.45	5516	31.45
2	7.240	24.970	181	5517	30.52	5517	30.52
3	7.060	25.060	177	4494	25.40	4494	25.40
4	6.800	25.580	174	4428	25.46	4428	25.46
Mean	**7.025**	**25.168**	**177**	**4989**	**28.21**	**4989**	**28.21**
Std Dev	**0.181**	**0.278**	**3**	**610**	**3.23**	**610**	**3.23**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	1.924	15.344	2691.671	2904			
2	1.909	13.435	2428.816	2853			
3	1.350	16.527	2923.978	3039			
4	1.324	11.568	2012.207	3218			
Mean	**1.627**	**14.219**	**2514.168**	**3004**			
Std Dev	**0.335**	**2.178**	**391.025**	**163**			


Load (N)
6000
5000
4000
3000
2000
1000
0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Extension (mm)
1
2
3
[4]

15%-425um

Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

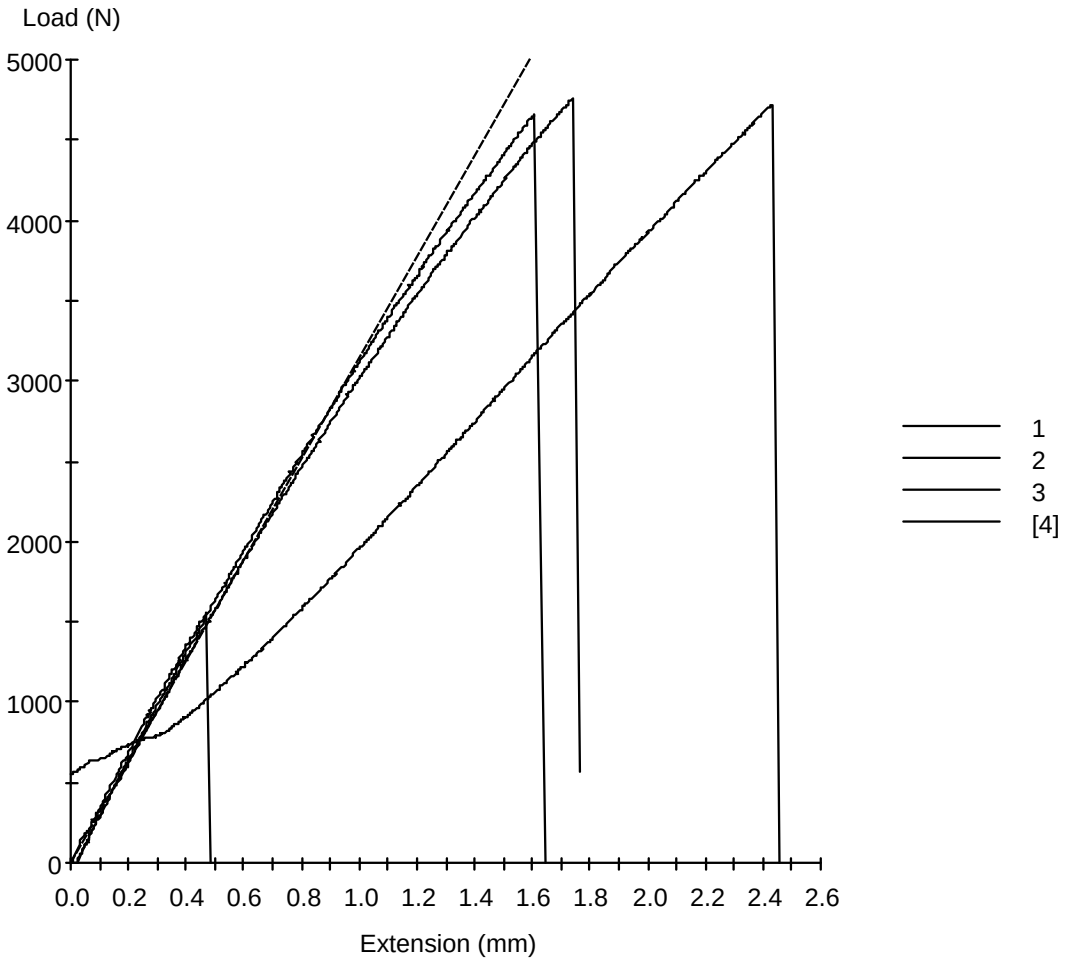
Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	6.800	25.270	172	4658	27.11	4658	27.11
2	6.330	25.280	160	4717	29.47	4717	29.47
3	6.400	25.010	160	1561	9.75	1561	9.75
4	6.260	25.340	159	4756	29.98	4756	29.98
Mean	**6.448**	**25.225**	**163**	**3923**	**24.08**	**3923**	**24.08**
Std Dev	**0.242**	**0.147**	**6**	**1575**	**9.63**	**1575**	**9.63**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	1.608	14.219	2443.251	2829			
2	2.431	26.962	4314.463	1841			
3	0.467	8.840	1414.991	3205			
4	1.738	14.244	2259.453	2970			
Mean	**1.561**	**16.066**	**2608.039**	**2711**			
Std Dev	**0.814**	**7.695**	**1222.554**	**600**			

Young's = 2829+3205+2970/3 = 3001

Tensile = 27.11+29.47+29.98= 28.85


Load (N)
5000
4000
3000
2000
1000
0
0.0 0.2 0.4 0.6 0.8 1.0 1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6
Extension (mm)
1
2
3
[4]

15%1180um

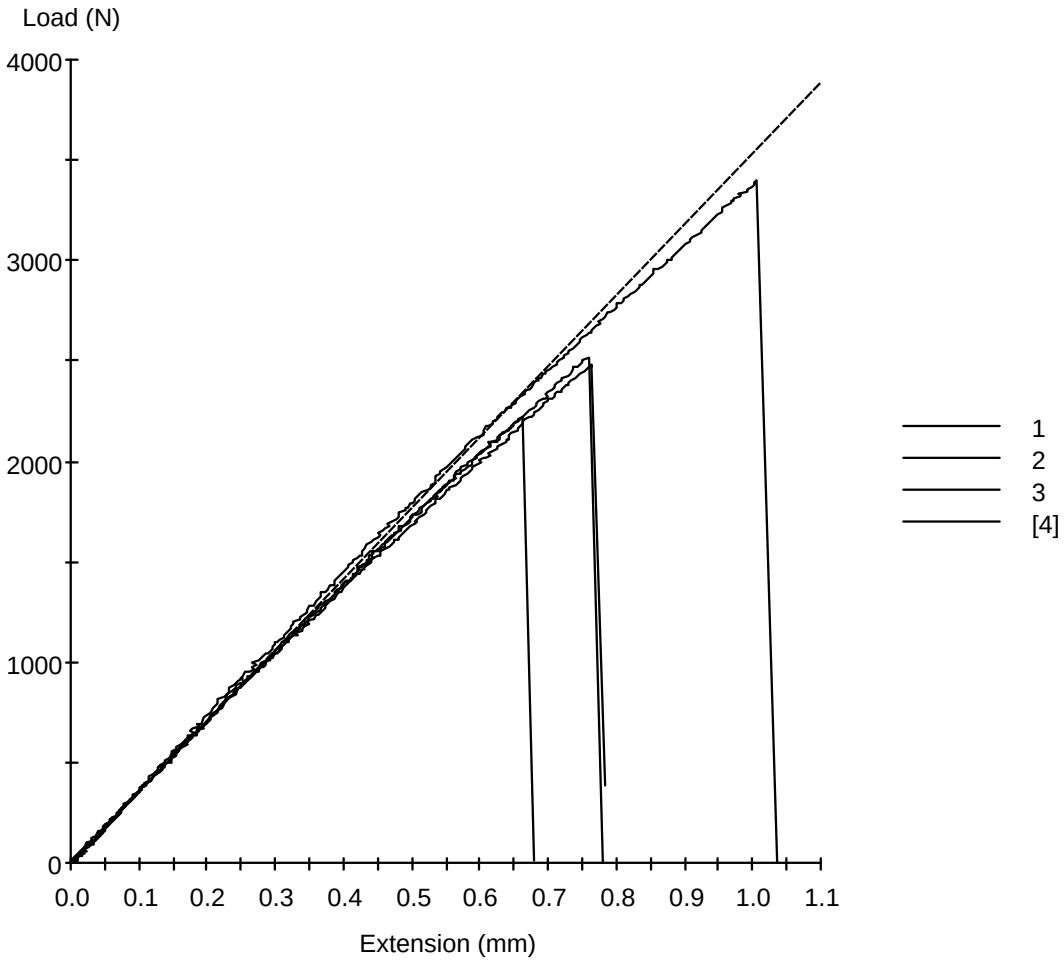
Report Date: 1/07/2010

Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.220	25.140	182	2518	13.87	2518	13.87
2	7.820	24.970	195	3399	17.41	3399	17.41
3	7.570	25.020	189	2222	11.73	2222	11.73
4	7.480	25.070	188	2484	13.25	2484	13.25
Mean	**7.522**	**25.050**	**188**	**2656**	**14.06**	**2656**	**14.06**
Std Dev	**0.248**	**0.073**	**6**	**513**	**2.40**	**513**	**2.40**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.760	9.118	1655.019	2927			
2	1.007	10.934	2135.075	2794			
3	0.663	7.845	1485.824	2825			
4	0.765	7.948	1490.524	2824			
Mean	**0.799**	**8.961**	**1691.610**	**2843**			
Std Dev	**0.146**	**1.436**	**305.932**	**58**			


Load (N)
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3000
2000
1000
0
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
1.1
Extension (mm)
1
2
3
[4]

16.7% 300 um

Report Date: 1/07/2010

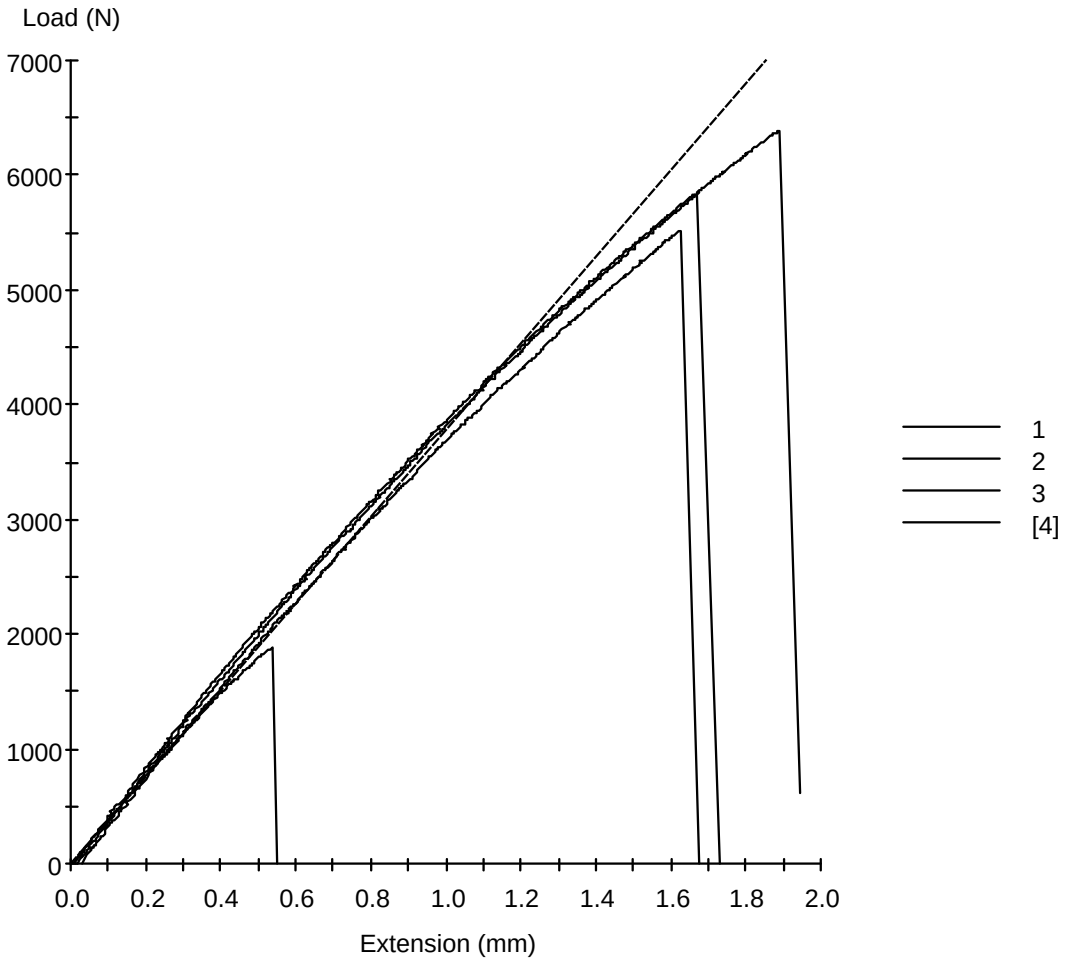
Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	8.150	24.990	204	6390	31.37	6390	31.37
2	7.870	25.290	199	5835	29.31	5835	29.31
3	7.560	25.200	191	1880	9.87	1880	9.87
4	7.530	25.150	189	5519	29.14	5519	29.14
Mean	**7.778**	**25.158**	**196**	**4906**	**24.92**	**4906**	**24.92**
Std Dev	**0.292**	**0.126**	**7**	**2049**	**10.09**	**2049**	**10.09**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	1.888	14.660	2985.748	2985			
2	1.667	14.573	2900.479	3002			
3	0.538	8.476	1614.734	2995			
4	1.628	17.521	3318.094	2994			
Mean	**1.430**	**13.807**	**2704.764**	**2994**			
Std Dev	**0.606**	**3.809**	**748.687**	**7**			

Tensile = 31.37 + 29.31 +29.14 /3 = 29.94


Load (N)
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4000
3000
2000
1000
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0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Extension (mm)
1
2
3
[4]

16.7% - 425um

Report Date: 7/07/2010

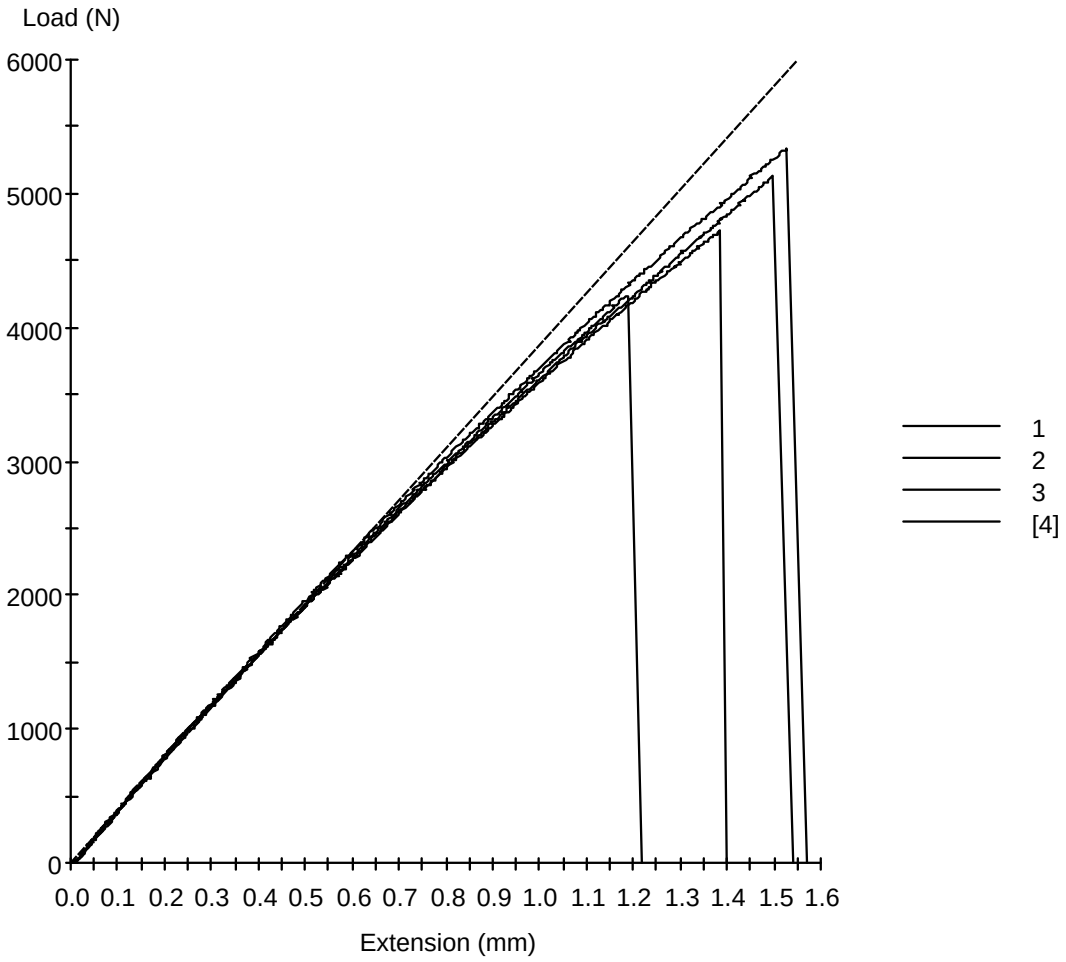
Test Date : 7/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	7.210	25.040	181	5334	29.55	5334	29.55
2	7.080	25.210	178	4717	26.43	4717	26.43
3	7.160	24.690	177	5126	29.00	5126	29.00
4	7.170	25.040	180	4239	23.61	4239	23.61
Mean	**7.155**	**24.995**	**179**	**4854**	**27.15**	**4854**	**27.15**
Std Dev	**0.054**	**0.219**	**2**	**484**	**2.72**	**484**	**2.72**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	1.524	13.927	2514.420	3250			
2	1.382	12.244	2185.430	3242			
3	1.496	13.980	2471.450	3258			
4	1.187	14.095	2530.534	3229			
Mean	**1.397**	**13.562**	**2425.458**	**3245**			
Std Dev	**0.153**	**0.881**	**161.950**	**12**			

Tensile = 29.55 + 26.43 + 29.00 = 28.32


Load (N)
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5000
4000
3000
2000
1000
0
0.0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6
Extension (mm)
1
2
3
[4]

16.7%1180um

Report Date: 1/07/2010

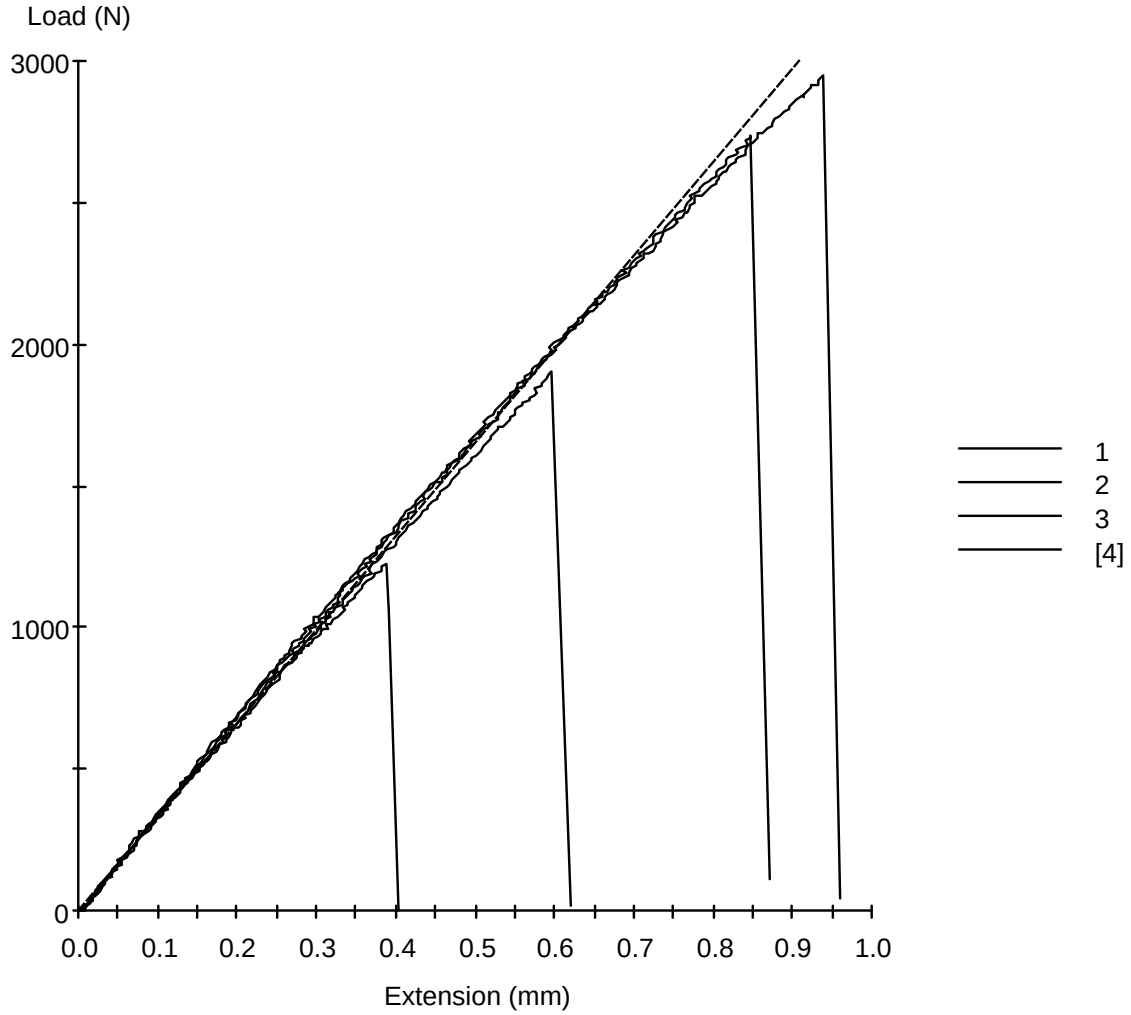
Test Date : 1/07/2010
Method : MMT Tensile Test with return.msm

Specimen Results:

Specimen #	Thickness mm	Width mm	Area mm^2	Peak Load N	Peak Stress MPa	Break Load N	Break Stress MPa
1	8.980	25.110	225	2950	13.08	2950	13.08
2	9.660	25.220	244	2735	11.23	2735	11.23
3	8.800	25.000	220	1222	5.55	1222	5.55
4	8.950	25.120	225	1900	8.45	1900	8.45
Mean	**9.098**	**25.112**	**228**	**2202**	**9.58**	**2202**	**9.58**
Std Dev	**0.383**	**0.090**	**10**	**795**	**3.29**	**795**	**3.29**

Specimen #	Elongation At Break mm	Stress At Offset Yield MPa	Load At Offset Yield N	Modulus MPa			
1	0.940	9.213	2077.334	2248			
2	0.848	8.075	1967.223	2116			
3	0.389	4.796	1055.116	2236			
4	0.598	7.606	1710.074	2207			
Mean	**0.693**	**7.422**	**1702.437**	**2202**			
Std Dev	**0.249**	**1.876**	**458.164**	**60**			

Tensile = 13.08 + 11.23 /2 = 12.155


Load (N)
3000
2000
1000
0
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
Extension (mm)
1
2
3
[4]

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